

Inorganic, Radioisotopic, and Organic Analysis of 241-AP-101 Tank Waste

S. K. Fiskum
P. R. Bredt
J. A. Campbell
O. T. Farmer
L. R. Greenwood
E. W. Hoppe
F. V. Hoopes
G. J. Lumetta
G. M. Mong
R. T. Ratner
C. Z. Soderquist
M. J. Steele
R. G. Swoboda
M. W. Urie
J. J. Wagner

August 2000

Prepared for BNFL, Inc.
under contract W375-LC-98-41698

PNWD-3046

LEGAL NOTICE

This report was prepared by Battelle Memorial Institute (Battelle) as an account of sponsored research activities. Neither Client nor Battelle nor any person acting on behalf of either:

MAKES ANY WARRANTY OR REPRESENTATION, EXPRESS OR IMPLIED, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, process, or composition disclosed in this report may not infringe privately owned rights; or

Assumes any liabilities with respect to the use of, or for damages resulting from the use of, any information, apparatus, process, or composition disclosed in this report.

References herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by Battelle. The views and opinions of authors expressed herein do not necessarily state or reflect those of Battelle.

Inorganic, Radioisotopic, and Organic Analysis of 241-AP-101 Tank Waste

S. K. Fiskum
P. R. Bredt
J. A. Campbell
O. T. Farmer
L. R. Greenwood
E. W. Hoppe
F. V. Hoopes
G. J. Lumetta
G. M. Mong
R. T. Ratner
C. Z. Soderquist
M. J. Steele
R. G. Swoboda
M. W. Urie
J. J. Wagner

August 2000

Prepared for
BNFL, Inc.
under contract W375-LC-98-41698

Battelle, Pacific Northwest Division
Richland, Washington 99352

Summary

Battelle received five samples from Hanford waste tank 241-AP-101, taken at five different depths within the tank. No visible solids or organic layer were observed in the individual samples. Individual sample densities were measured, then the five samples were mixed together to provide a single composite. The composite was homogenized and representative sub-samples taken for inorganic, radioisotopic, and organic analysis. All analyses were performed on triplicate sub-samples of the composite material.¹ The sample composite did not contain visible solids or an organic layer. A subsample held at 10°C for seven days formed no visible solids.

The characterization of the 241-AP-101 composite samples included:

- Inductively-coupled plasma spectrometry for Ag, Al, Ba, Bi, Ca, Cd, Cr, Cu, Fe, K, La, Mg, Mn, Na, Nd, Ni, P, Pb, Pd, Ru, Rh, Si, Sr, Ti, U, Zn, and Zr
(Note: Although not specified in the test plan, As, B, Be, Co, Li, Mo, Sb, Se, Sn, Tl, V, W, and Y were also measured and reported for information only)
- Radioisotopic analyses for total alpha and total beta activities, ³H, ¹⁴C, ⁶⁰Co, ⁷⁹Se, ⁹⁰Sr, ⁹⁹Tc as pertechnetate, ¹⁰⁶Ru/Rh, ¹²⁵Sb, ¹³⁴Cs, ¹³⁷Cs, ¹⁵²Eu, ¹⁵⁴Eu, ¹⁵⁵Eu, ²³⁸Pu, ²³⁹⁺²⁴⁰Pu, ²⁴¹Am, ²⁴²Cm, and ²⁴³⁺²⁴⁴Cm
- Inductively-coupled plasma mass spectrometry for ²³⁷Np, ²³⁹Pu, ²⁴⁰Pu, ⁹⁹Tc, ¹²⁶Sn, ¹²⁹I, ²³¹Pa, ²³³U, ²³⁴U, ²³⁵U, ²³⁶U, ²³⁸U, ²⁴¹AMU, ²⁴²AMU, ²⁴³AMU, As, B, Be, Ce, Co, Cs, Eu, I, Li, Mo, Pr, Rb, Sb, Se, Ta, Te, Th, Tl, V, and W
- Total U by kinetic phosphorescence analysis
- Ion chromatography for Cl, F, NO₂, NO₃, PO₄, SO₄, acetate, formate, oxalate, and citrate
- Density, inorganic carbon and organic carbon by two different methods, mercury, free hydroxide, ammonia, and cyanide
- Polychlorinated biphenyls

The 241-AP-101 composite met all contract limits (molar ratio of analyte to sodium or ratio of becquerels of analyte to moles of sodium) defined in Specification 7 for Envelope A.² Except for a few cases, the characterization results met or surpassed the quality control requirements established by the governing quality assurance plan and met or surpassed the minimum reportable quantity requirements specified by BNFL.

¹ Specific inorganic, radiochemical, and organic analytes of interest and reporting requirements were defined in CCN: 01260 Letter to Eugene Morrey and translated to the laboratory personnel via ASR 5778, test instructions BNFL-TP-29953-81 and -83.

² As provided by Tank Waste Remediation System Privatization Contract DE-AC27-96-RL13308, Mod 14 (4/18/00) Table TS-7.1 LAW Chemical Composition, Soluble Fraction Only and Table TS-7.2 LAW Radionuclide Content, Soluble Fraction Only.

Terms and Abbreviations

AEA	alpha energy analysis
ASR	analytical services request
AMU	atomic mass units
BNFL	BNFL, Inc; subsidiary of British Nuclear Fuels, Ltd.
CCV	continuing calibration verification
COC	chain of custody
CVAA	cold vapor atomic absorption
EQL	estimated quantitation level
GEA	gamma energy analysis
HASQARD	Hanford analytical services quality assurance requirements document
IC	ion chromatography
ICP	inductively coupled plasma/atomic emission spectrometry
ICP/MS	inductively coupled plasma/mass spectrometry
IDL	instrument detection limit
ISE	ion specific electrode
KPA	kinetic phosphorescence analyzer
LCS	laboratory control standard
MDL	method detection limit
MRQ	minimum reportable quantity
MSA	method of standard addition
NA	not applicable
NIST	National Institute of Standards and Technology
NP	not performed
PCB	polychlorinated biphenyl
QA	quality assurance
QC	quality control
RPD	relative percent difference
RSD	relative standard deviation
SAL	Shielded Analytical Laboratory
SBMS	standards based management system
SRM	Standard Reference Material
TC	total carbon
TCLP	toxicity characteristic leaching procedure
TIC	total inorganic carbon
TOC	total organic carbon
TRU	transuranic

Units

°C - °F	degrees Centigrade / degrees Fahrenheit
Bq	becquerel (disintegrations per second)
g	gram
g/mL	gram per milliliter
µg/mL	microgram per milliliter
µCi/mL	microcurie per milliliter
mL	milliliter
mmole/mL	millimole per milliliter
M	molarity, moles per liter

Contents

1.0	INTRODUCTION	1.1
2.0	INITIAL SAMPLE PROCESSING	2.1
2.1	Sample Density and Homogenization.....	2.1
2.2	Sample Digestion for Analysis.....	2.1
2.3	Sample Precipitation at Reduced Temperature	2.3
2.4	Polychlorinated Biphenyl Extraction.....	2.3
2.5	Remaining AP-101 Composite Material.....	2.3
3.0	ANALYTICAL RESULTS.....	3.1
3.1	Analyte List Modifications	3.1
3.2	Data Limitations	3.2
3.3	General Observations.....	3.2
4.0	PROCEDURES, QUALITY CONTROL, AND DATA EVALUATION.....	4.1
4.1	Metals Analysis by ICP—Tables 3.1 and 4.1	4.1
4.2	Total Uranium (KPA) Tables 3.1 and 4.1	4.2
4.3	Radioisotopic Analyses, Tables 3.2 and 4.2	4.2
4.4	Inductively-Coupled Plasma—Mass Spectrometry, Tables 3.3. and 4.3	4.5
4.5	Anion Analysis, Tables 3.4 and 4.4.....	4.5
4.6	Miscellaneous Analytes, Tables 3.5 and 4.5	4.7
5.0	REFERENCES.....	5.1
	APPENDIX A: TANK 241-AP-101 GRAB SAMPLES	A.1
	APPENDIX B: TEST INSTRUCTIONS	B.1
	APPENDIX C: ANALYTICAL REPORTS	C.1

Figures

Figure 2.1. Sample Processing	2.2
-------------------------------------	-----

Tables

Table 2.1. 241-AP-101 Sample Densities and Volumes.....	2.1
Table 3.1. 241-AP-101 Composite, ICP Metals Results	3.4
Table 3.2. 241-AP-101 Composite, Radionuclide Results	3.5
Table 3.3. 241-AP-101 Composite, ICP-MS Stable Element Results.....	3.6
Table 3.4. 241-AP-101 Composite, IC Results.....	3.7
Table 3.5. 241-AP-101 Composite, Miscellaneous Analyte Results.....	3.8
Table 4.1. 241-AP-101 Composite, ICP Metals QC Results	4.11
Table 4.2. 241-AP-101 Composite, Radionuclide QC Results	4.12
Table 4.3. 241-AP-101 Composite, ICP-MS Stable Element QC Results.....	4.13
Table 4.4. 241-AP-101 Composite, IC QC Results.....	4.14
Table 4.5. 241-AP-101 Composite, Miscellaneous Analyte QC Results	4.15

1.0 Introduction

This report presents the inorganic, organic and radioisotopic analytical results for a composite sample obtained from tank 241-AP-101 (AP-101). This work was conducted in response to a request by BNFL.³ The results of the analyses are used to assess the waste composition relative to the contract limits defined in Specification 7 for envelope A.

Hanford waste tank 241-AP-101 was sampled on February 8, 2000 from Riser 002 at depths of 10, 100, 190, 290, and 400 inches from the tank bottom, collecting nominally 130 mL per sample.⁴ These samples were received under chain-of-custody by the Radiochemical Processing Laboratory Shielded Analytical Laboratory. All samples were clear yellow with no visible settled or suspended solids. The AP-101 grab samples were mixed to form one large composite sample according to Test Plan BNFL-TP-29953-083. Sub-samples from this homogenized AP-101 composite sample were then delivered to various laboratories for specific analyses as defined in the analytical service request (ASR) 5778. The sample was given an internal tracking number of 00-1701. All analyses were run in triplicate.

The PNNL standards based management system (SBMS) quality assurance plan was used in support of all analytical operations and is in compliance with HASQARD. The inorganic, radioisotopic, and organic analytes of interest, recommended methods, detection limits, and quality assurance parameters were defined by BNFL. The quality requirements were included in ASR 5778. Analyte determinations were performed according to project-approved procedures.

This report presents the physical observations from AP-101 and individual density measurements from the various tank depths sampled. Precipitate production was evaluated at 10°C for seven days on a composite subsample. Also presented are the inorganic, radioisotopic, and organic analytical results for the triplicate AP-101 composite samples. Analyte results are compared to Specification 7 Envelope A limits, where applicable. Data limitations are also described. Quality control, detection limits, and other quality control indicators are discussed relevant to the reporting method.

Revision 1 includes addition of PCB results as well as minor editorial corrections.

³ Statement of Work for 241-AP-101 Samples. Transmitted via letter: ME Johnson to EV Morrey, Contract No. DE-AC06-96RL13308-W375-Request for Proposal to Conduct Revised Analysis of Tank 241-AP-101 Samples, CCN: 012160.

⁴ Wood, R. F., Letter Report, CH2M Hill, Hanford Group, Inc., to J.J. Short, DOE-ORP, "Sample Management Document Package for Grab Samples form Tank 241-AP-101," Letter No. CHG-0000767 dated February 15, 2000.

2.0 Initial Sample Processing

2.1 Sample Density and Homogenization

The samples from the five different sampling depths were confirmed to be clear and yellow with no suspended or settled solids, as indicated on the chain-of-custody form. Each sample density was taken by determining the net sample mass in a 25-mL glass volumetric flask.⁵ The individual densities, given in Table 2.1, show a slight decrease with increasing sampling elevation.

Table 2.1. 241-AP-101 Sample Densities and Volumes

<i>Sample ID</i>	1AP-00-1	1AP-00-4	1AP-00-7	1AP-00-10	1AP-00-13
<i>Elevation (inches)*</i>	400	290	190	100	10
<i>Density (g/mL)</i>	1.290	1.291	1.291	1.320	1.328
<i>Mass received (g)</i>	174.3	170.2	169.7	173.6	172.2
<i>Volume received (mL)</i>	135.2	131.8	131.5	131.6	129.7

*Elevation is the distance from the tank bottom to the mouth of the sample bottle.

The entire contents of each sample container were transferred to a 1-L jar; the net mass transferred was calculated by difference from the full and empty container masses.⁶ The 860-g (659.8-mL) AP-101 composite was sealed and stirred for 54 minutes using a magnetic stir bar. A 101-mL sub-sample was transferred to a glass bottle and sealed. For subsequent precipitation/crystallization testing at reduced temperature. Portions of the remaining composite material were used for subsequent analyses. The composite sample did not show signs of phase separation or precipitation. The overall sample processing is summarized in Figure 2.1.

2.2 Sample Digestion for Analysis

After sample homogenization, the AP-101 composite sub-samples were delivered directly to the laboratories for various measurements including mercury, cyanide, hydroxide, tritium, total organic carbon, total inorganic carbon, ammonia, ion chromatography (inorganic and organic ions), ¹⁴C, pertechnetate, ⁷⁹Se, and gamma spectrometry. The AP-101 composite density (see Table 3.5) was determined in the Shielded Analytical Laboratory (SAL).

The SAL processed 5-mL aliquots in triplicate according to PNL-ALO-128, HNO₃-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater, for subsequent ICP and ICP/MS analyses. The acid extracted solutions were brought to a nominal 25-mL volume and absolute volumes determined based on final solution weights and densities. This acid digestion resulted in a solution with a small amount of white floating particulates. The floating particulates were removed by filtration. The filtrates were analyzed by ICP and ICP/MS. Along with the samples, the SAL processed a reagent blank, a toxicity characteristic leach protocol (TCLP)-metals spiked blank, and TCLP-metals spiked sample. The TCLP metals spike included Ag, As, Ba, Cd, Cr, Ni, Pb, Se, Tl, and Zn.

⁵ PNNL Test Instruction Number 29953-81, Rev. 0, Density Measurements on As-Received AP-101 Samples, Paul Bredt, 2/10/00

⁶ PNNL Test Instruction Number BNFL-TP-29953-83, Rev. 0, "AP-101 Homogenization and Subsampling," by Paul Bredt, 3/31/00

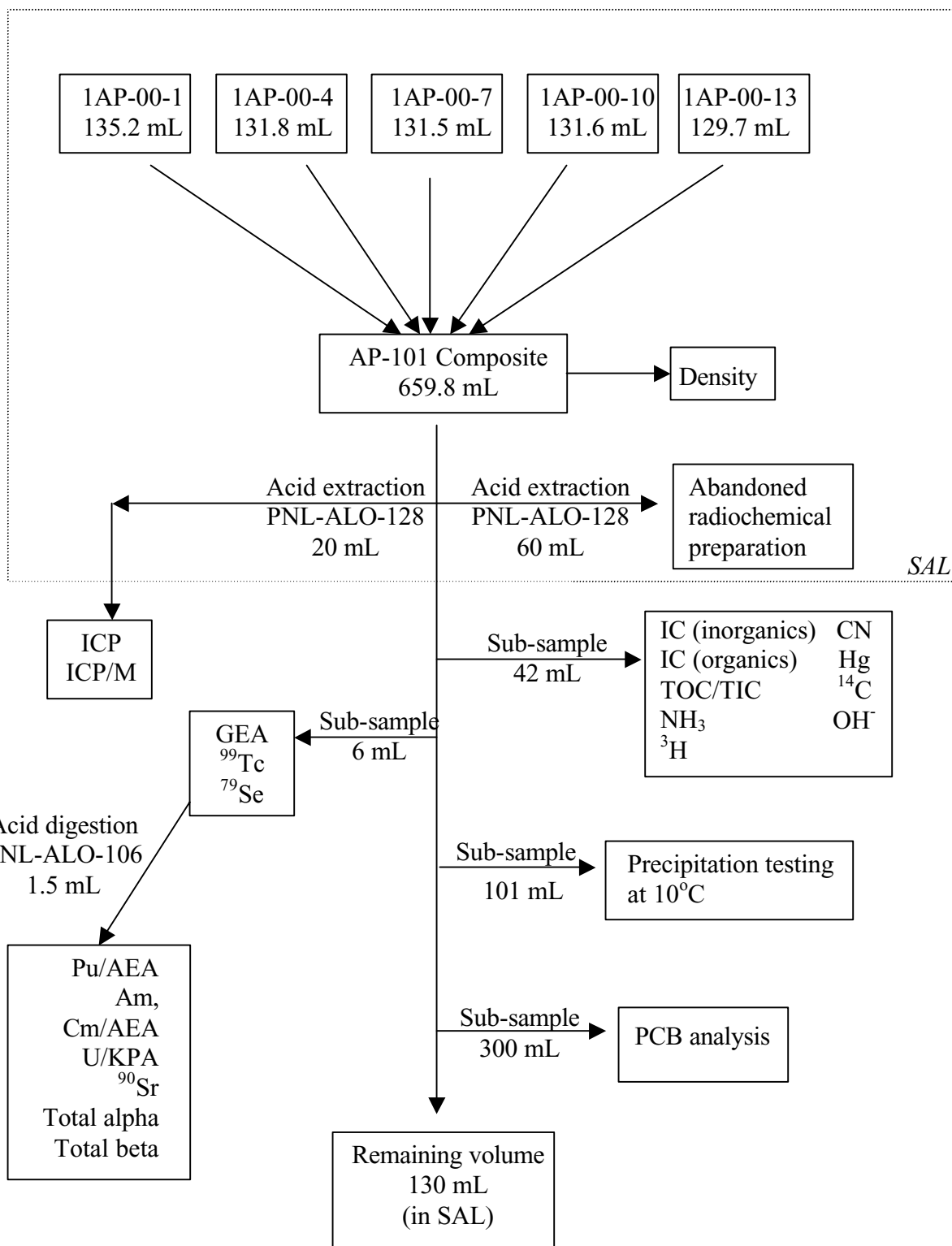


Figure 2.1. 241-AP-101 Sample Processing

The SAL also processed 20-mL AP-101 composite aliquots in triplicate according to PNL-ALO-128. The acid extraction was insufficient to neutralize the 20-mL sample and it resulted in a small amount of solid residue formation (settled on the vial bottom as opposed to floating particulates). Additional 8M HNO₃ was added to one aliquot to dissolve the residual solids. This resulted in severe foaming as well as visible NO_x production. Continued 8M HNO₃ addition resulted in mixed-phases consisting of whitish floating material (estimated volume of between 0.1 and 0.5 cubic centimeters) on top of a near colorless solution. Acid digestion/neutralization of the three 20-mL aliquots of AP-101 composite was abandoned in favor of performing a nitric acid digestion on 0.5-mL aliquots according to PNL-ALO-106, Acid Digestion for Preparation of Samples for Radiochemical Analysis. Nitric acid additions and heating continued until all organic/nitric reactions had visibly ceased. The samples were then brought to volume in 10-mL volumetric flasks with 2M HNO₃. If insoluble material formed, it was too small to discern. A reagent blank was run with the samples. These digestions were used for the subsequent analyses of total alpha, total beta, ²³⁹⁺²⁴⁰Pu, ²³⁸Pu, ²⁴¹Am, ²⁴²Cm, ²⁴³⁺²⁴⁴Cm, total U by kinetic phosphorescence analysis (KPA), and ⁹⁰Sr. A laboratory control sample (LCS) was not available for radiochemical analyses. Post digestion blank spikes and matrix spikes were prepared at the time of radiochemical separation.

2.3 Sample Precipitation at Reduced Temperature

A 101-mL aliquot of the AP-101 composite was visually inspected using a video camera installed in the HLRF hot cell. The sample contained a very small amount of particulate material. This was removed by filtering through a 0.45-μm nylon membrane. The clarified liquid was transferred to a clean glass jar and was then placed in a cooling bath at 10°C. The sample was inspected daily (excluding weekends) for a period of seven days. No solids formed after seven days. Thus, the AP-101 solution appears to be stable towards precipitation at 10°C.

2.4 Polychlorinated Biphenyl Extraction

The AP-101 composite was sampled and extracted for polychlorinated biphenyl (PCB) analysis according to special instructions in ASR 5778 and test plan BNFL-TP-29953-089. Sub-samples consisted of duplicate 100-mL aliquots (surrogate spike only) and duplicate 50-mL aliquots (surrogate and Aroclor spike); a blank consisting of water (surrogate spike only) was processed with the sample set. All samples were extracted three times with 25-mL methylene chloride. The extracts were combined and transferred from the SAL to the PCB workstation under COC. The methylene chloride extracts were dried over anhydrous sodium sulfate and concentrated to a 2-mL volume. Additional extract cleanup was performed following exchange into hexane. The hexane residues were then analyzed for PCBs according to BNFL-TP-29953-027 by gas chromatography.

2.5 Remaining AP-101 Composite Material

The combined total volume sampled for the various analyses was 529-mL. The amount remaining after all sampling events were completed is nominally 130-mL or 171-g.

3.0 Analytical Results

Tables 3.1 through 3.5 provide inorganic, radioisotopic, and organic analytical results for the triplicate AP-101 composite samples. The average of the triplicate values and the relative standard deviation (RSD) are also given. Results are reported in $\mu\text{g/mL}$ or $\mu\text{Ci/mL}$. The reference date for radioisotope activities is the analysis date. The nominal propagated uncertainties are also provided as $1-\sigma$ unless otherwise noted.

Preparation blank results are also reported, as appropriate. Several analytes were measured directly, requiring no preparation blank (e.g., GEA, IC, and hydroxide). In these instances, the preparation blank field is empty. Generally, analyte concentrations in the preparation blanks were either insignificant relative to the sample analyte concentration or at or near the method detection limit. This indicates processing steps did not result in significant sample contamination (except in the case of B, see discussion in section 3.2).

Specification 7 for Envelope A defines limits for several analytes relative to sodium concentration (moles analyte per mole Na or Bq analyte per mole Na). These ratio limits are provided in conjunction with as-measured ratios. In all cases the mole or Bq analyte to moles Na ratio did not exceed the limits defined in Specification 7 for Envelope A. It should be noted the K concentration is also relatively high (0.8M) and may also affect other process steps including Cs ion exchange, Tc ion exchange, and vitrification.

Specific quality control and quality assurance discussions are given in Section 4.0.

3.1 Analyte List Modifications

The analyte list provided by the client was extracted from Contract Specification 7. Several modifications to this analyte list had to be incorporated as follows:

- The Pu separation and subsequent analysis by AEA requires ^{239}Pu and ^{240}Pu be measured together as a sum, not as separate isotopes. The alpha energy peaks from these isotopes are non-resolvable. The lower of the two MRQs for these analytes was used for compliance purposes.
- The laboratory was directed to determine pertechnetate ($^{99}\text{TcO}_4^{-+7}$) using separations and beta counting techniques, as opposed to total ^{99}Tc . The procedure was modified slightly to exclude the sample oxidation step so that the nonpertechnetate fraction was not oxidized. Also, instead of measuring the ^{99}Tc by liquid scintillation, sample preparations were counted with gas-flow proportional counters.
- Analysis for ^{135}Cs was not performed as requested by gamma spectrometry. There are no gamma-emissions associated with this isotope.
- Analyte concentrations, in addition to the required and opportunistic analytes, are provided in Tables 3.1 and 3.4. These additional analytes were measured as part of the method and are provided for additional information.
- Total carbon was measured using the furnace oxidation method as opposed to individual measurements of TOC and TIC. This method is considered reliable for only total carbon.

3.2 Data Limitations

- The reported fluoride results, average 2900 µg/mL, represent the summation of fluoride, acetate, and formate concentrations, as these are not resolvable on the inorganic anion analysis IC system. The acetate and formate were quantified on the organic anion IC system. The sum of these two analytes is 2840 µg/mL. This indicates very little fluoride is present in AP-101.
- The reported acetate results represent the summation of glycolate and acetate, as these are not resolvable on the organic IC system.
- ICP-MS analytes at AMU-241, 242, and 243, were measured relative to a ²³⁹Pu standard and the reported results are considered semi-quantitative. They are listed in the stable element data Table 3.3. with concentration units of µg/mL. Although these elements are unstable, an activity concentration cannot be applied without knowing the specific activity, and thus the analyte identity. For example, if ²⁴¹AMU is assigned to Am, then the activity becomes <0.038 µCi/mL; if ²⁴¹AMU is assigned to Pu, then the activity becomes <1.2 µCi/mL.
- The ¹²⁶Sn and ²³¹Pa ICP-MS analytes were determined relative to related isotopes and the reported results are considered semi-quantitative.
- Boron was present in the preparation blank at 40% of the sample concentration indicating the sample boron concentration could be biased high by the digestion method.
- Arsenic was present in the preparation blank at 10% of the sample concentration indicating the sample arsenic concentration could be biased high by the digestion method.
- The alpha summation is a better representation of the total alpha activity than the total alpha determination for this matrix. Solids loading on the total alpha mount resulted in some alpha attenuation.
- The acid digestion preparations for ICP and ICP-MS were filtered to remove a small amount of floating particulates. This leaves open the possibility that one or more analyses could be biased low.
- PCBs have low solubilities and tend to plate out on vessel walls. Typically sample vessels are rinsed with a PCB solvent (methylene chloride) to remove potentially plated PCBs. No vessel rinsing was performed during the sampling, homogenization, or sub-sampling steps. This could cause a low bias for the PCB results.
- The reported total PCB MDL represents the MDL summation of seven Aroclors, where each Aroclor MDL is 0.2 µg/L.

3.3 General Observations

- The total ⁹⁹Tc measured by ICP-MS agreed well with the pertechnetate analysis (separations and beta counting), indicating the ⁹⁹Tc in AP-101 is present in the pertechnetate form.

- The U concentrations determined by KPA and ICP-MS agreed within the 2- σ uncertainty.
- The total beta activity is equivalent to the ^{137}Cs activity indicating ^{137}Cs is the primary β -emitting isotope in this waste.
- The phosphorous concentration is virtually identical to the phosphate concentration indicating the phosphorous is primarily present as phosphate.
- Generally the analyte concentrations measured by both ICP-MS and ICP were consistent.

Table 3.1. 241-AP-101 Composite, ICP Metals Results

Lab ID	PB-1701	00-1701	00-1701D	00-1701T				Measured	Specification 7
Sample ID	prep blank	sample	duplicate	triplicate				Mole analyte	Envelope A
Run date	4/11/00	4/11/00	4/11/00	4/11/00	Average	RSD	Average	per mole Na	Limits
Units	µg/mL	µg/mL	µg/mL	µg/mL	µg/mL	%	M	Ratio	Mole analyte
									per mole Na
									Ratio
Analyte ⁽¹⁾									
Ag	<0.12	<0.63	<0.63	<0.62	<0.63				
Al	4.5	7,380	6,680	6,760	6,940	5.5	2.6E-1	4.6E-2	2.5E-1
Ba	<0.05	[0.32]	[0.34]	[0.33]	[0.33]	3.0	[2.4E-6]	[4.3E-7]	1.0E-4
Bi	<0.48	<2.5	<2.5	<2.5	<2.5				
Ca	[1.4]	[7.8]	[7.5]	[7.7]	[7.7]	2.0	[1.9E-4]	[3.4E-5]	4.0E-2
Cd	<0.07	[2.0]	[1.9]	[2.0]	[2.0]	2.9	[1.7E-5]	[3.1E-6]	4.0E-3
Cr	<0.1	158	137	135	143	8.9	2.8E-3	4.9E-4	6.9E-3
Cu	[0.18]	[1.7]	[1.5]	[1.5]	[1.6]	7.4			
Fe	[0.38]	[2.9]	[2.4]	[2.2]	[2.5]	14	[4.5E-5]	[8.0E-6]	1.0E-2
K	<9.7	31,700	30,500	31,500	31,200	2.1	8.0E-1	1.4E-1	1.8E-1
La	<0.24	<1.3	<1.3	<1.2	<1.3		<9.4E-6	<1.7E-6	8.3E-5
Mg	<0.48	<2.5	<2.5	<2.5	<2.5				
Mn	<0.24	<1.3	<1.3	<1.2	<1.3				
Na	14	132,000	125,000	131,000	129,300	2.9	5.62E+0		
Nd	<0.48	<2.5	<2.5	<2.5	<2.5				
Ni	1.6	8.6	7.7	7.5	7.9	7.6	1.4E-4	2.4E-5	3.0E-3
P	<0.48	371	290	281	314	16	1.0E-2	1.8E-3	3.8E-2
Pb	<0.48	[15]	[15]	[15]	[15]	0	[7.2E-5]	[1.3E-5]	6.8E-4
Pd	<3.6	<19	<19	<19	<19				
Rh	<1.5	<7.6	<7.6	<7.5	<7.6				
Ru	<5.3	<28	<28	<27	<28				
Si	[24]	143	132	137	137	4.0			
Sr	<0.07	<0.38	<0.38	<0.37	<0.38				
Ti	<0.12	<0.63	<0.63	<0.62	<0.63				
U	<9.7	[68]	[62]	[56]	[62]	9.7	[2.6E-4]	[4.6E-5]	1.2E-3
U(KPA) ⁽²⁾	0.0054	51.7	51.2	50.3	51.1	1.4	2.1E-4	3.8E-5	1.2E-3
U(ICP-MS) ⁽³⁾	<0.14	58.9	52.9	54.1	55.3	5.7	2.3E-4	4.1E-5	1.2E-3
Zn	[0.91]	[5.8]	[5.5]	[5.4]	[5.6]	3.7			
Zr	<0.24	[1.4]	[1.4]	[1.3]	[1.4]	4.2			
Additional analyte information									
As*	<1.21	<6.3	<6.3	<6.2	<6.3				
Be*	<0.05	[1.1]	[1.0]	[1.0]	[1.0]	5.6			
B*	6.62	15	14	15	15	2.6			
Co*	<0.24	<1.3	<1.3	<1.2	<1.3				
Li*	<0.15	<0.76	<0.76	<0.75	<0.76				
Mo*	<0.24	[12]	[10]	[9.8]	[11]	11			
Se*	<1.2	<6.3	<6.3	<6.2	<6.3				
Sn	<7.3	[60]	<38	<37	<38				
Tl*	<2.4	<13	<13	<12	<13				
V*	<0.24	<1.3	<1.3	<1.2	<1.3				
W*	<9.7	<51	<51	<50	<51				
Y	<0.24	<1.3	<1.3	<1.2	<1.3				

(1) Overall error for reported results is estimated to be within +/-15%, 2-σ; however results in brackets "[]" are less than the estimated quantitation level (i.e., 10-times MDL) and error is anticipated to exceed +/-15%.

(2) U (KPA) indicates U determination by kinetic phosphorescence on 4/26/00 with an uncertainty of 3%, 1-σ.

(3) U (ICP-MS) indicates opportunistic U determination by ICP-MS on 4/18/00, with an uncertainty of 3.5%, based on one standard deviation triplicate sample summing all U isotopes.

*Analytes on ICP-MS Specification 7 list.

Table 3.2. 241-AP-101 Composite, Radionuclide Results

Lab ID	PB-1701	00-1701	00-1701D	00-1701T	Propagated				Measured	Specification 7
Sample ID	prep blank ⁽¹⁾	sample	duplicate	triplicate	1-σ	Average	RSD	Analysis	Bq analyte	Envelope A
Units	μCi/mL	μCi/mL	μCi/mL	μCi/mL	% Error ⁽³⁾	μCi/mL	%	Date	per mole Na	Limits
									Ratio	Bq analyte
										per mole Na
										Ratio
Analyte										
³ H	<2E-4	5.05E-3	4.84E-3	4.60E-3	3	4.83E-3	4.7	4/16-21/00		
¹⁴ C	<4E-5	2.56E-4	2.55E-4	2.77E-4	8	2.63E-4	4.7	5/11/00		
⁶⁰ Co (GEA)		3.22E-3	3.40E-3	3.24E-3	7	3.29E-3	3.0	4/28-30/00	2.2E+4	6.1E+4
⁷⁹ Se	<3E-6	5.56E-5	6.97E-5	4.93E-5	5-11	5.82E-5	18	5/18/00		
⁹⁰ Sr	<2E-4	8.71E-2	8.77E-2	8.75E-2	3	8.74E-2	0.3	5/1/00	5.8E+5	4.4E+7
⁹⁹ Tc (+7)	<2E-5	4.71E-2	4.34E-2	5.10E-2	4	4.72E-2	8.1	4/24-25/00	3.1E+5	7.1E+6
¹⁰⁶ Ru/Rh (GEA)		<9E-2	<9E-2	<9E-2		<9E-2		4/28-30/00		
¹²⁵ Sb (GEA)		<6E-2	<6E-2	<6E-2		<6E-2		4/28-30/00		
¹³⁴ Cs (GEA)		4.77E-2	4.81E-2	4.62E-2	3	4.73E-2	2.1	4/28-30/00		
¹³⁷ Cs (GEA)		1.44E+2	1.44E+2	1.45E+2	2	1.44E+2	0.4	4/28-30/00	9.5E+8	4.3E+9
¹⁵² Eu (GEA)		<2E-3	<3E-3	<2E-3		<2E-3		4/28-30/00		
¹⁵⁴ Eu (GEA)		<2E-3	<2E-3	<2E-3		<2E-3		4/28-30/00	<3E+5 ⁽⁴⁾	1.2E+6 ⁽⁴⁾
¹⁵⁵ Eu (GEA)		<4E-2	<4E-2	<4E-2		<4E-2		4/28-30/00		
²³⁸ Pu	<2E-7	1.39E-5	1.49E-5	1.54E-5	6	1.47E-5	5.2	4/25-26/00		
²³⁹ Pu + ²⁴⁰ Pu	<2E-7	1.24E-4	1.28E-4	1.27E-4	4	1.26E-4	1.6	4/25-26/00		
²⁴¹ Am (GEA)		<4E-2	<4E-2	<4E-2		<4E-2		4/28-30/00		
²⁴¹ Am	<5E-7	1.73E-4	1.65E-4	1.70E-4	5	1.69E-4	2.4	4/25-26/00		
²⁴² Cm	<8E-8	<1E-7	<9E-8	<2E-7		<2E-7		4/25-26/00		
²⁴³ + ²⁴⁴ Cm	<8E-8	1.34E-6	7.40E-7	1.02E-6	21	1.03E-6	29.1	4/25-26/00		
Total alpha	<7E-5	2.55E-4	2.22E-4	2.65E-4	14	2.47E-4	9.1	4/24/00		
Alpha sum ⁽²⁾		3.12E-4	3.09E-4	3.13E-4		3.11E-4	0.7	4/25-26/00	2.0E+3	4.8E+5
Total beta	8.57E-5	1.46E+2	1.40E+2	1.54E+2	4	1.47E+2	4.8	4/20-24/00		
ICP-MS Analytes										
⁹⁹ Tc (total)	<2.5E-4	5.14E-2	4.76E-2	4.80E-2	1-4	4.90E-2	4.3	4/13/00	3.2E+5	7.1E+6
¹²⁶ Sn	<1.3E-3	<8.6E-4	<8.6E-4	<8.2E-4		<8.6E-4		4/19/00		
¹²⁹ I	<2.5E-6	7.72E-5	7.73E-5	8.71E-5	3-7	8.05E-5	7.1	4/17/00		
²³¹ Pa	<1.0E-4	<1.0E-4	<1.1E-4	<1.1E-4		<1.1E-4		4/19/00		
²³³ U	<8.8E-6	5.50E-5	5.60E-5	4.80E-5	7-11	5.30E-5	8.2	4/13/00		
²³⁴ U	<5.7E-6	2.70E-5	2.40E-5	2.10E-5	12-21	2.40E-5	12.5	4/13/00		
²³⁵ U	<6.8E-9	1.06E-6	9.94E-7	9.82E-7	0.3	1.01E-6	4.2	4/13/00		
²³⁶ U	<5.9E-8	2.23E-6	2.07E-6	2.08E-6	2-6	2.13E-6	4.2	4/13/00		
²³⁸ U	<4.7E-8	1.95E-5	1.76E-5	1.80E-5	0.5	1.84E-5	5.5	4/13/00		
²³⁷ Np	<5.3E-6	<5.4E-6	<5.4E-6	<5.5E-6		<5.5E-6		4/19/00		
²³⁹ Pu	<1.2E-3	<1.2E-3	<1.2E-3	<1.2E-3		<1.2E-3		4/19/00		
²⁴⁰ Pu	<2.4E-3	<2.4E-3	<2.4E-3	<2.4E-3		<2.4E-3		4/19/00		

(1) Samples run directly by GEA and did not require a preparation blank.

(2) Alpha sum refers to the summation of ²³⁸Pu, ²³⁹+²⁴⁰Pu, ²⁴¹Am, and ²⁴³+²⁴⁴Cm alpha activities.

(3) The error represents the error of each reported sample concentration. In some cases, the error was identical and a single error value is given. In other cases, the reported values had varying errors and the error range is given.

(4) Based on the summation of ¹⁵⁴Eu and ¹⁵⁵Eu.

Table 3.3. 241-AP-101 Composite, ICP-MS Stable Element Results

<i>Lab ID</i>	PB-1701	00-1701	00-1701D	00-1701T			
<i>Sample ID</i>	prep blank	sample	duplicate	triplicate	Propagated	Average	RSD
<i>Run date</i>	4/13-19/00	4/13-19/00	4/13-19/00	4/13-19/00	1- σ		
<i>Units</i>	$\mu\text{g/mL}$	$\mu\text{g/mL}$	$\mu\text{g/mL}$	$\mu\text{g/mL}$	% Error ⁽¹⁾	$\mu\text{g/mL}$	%
Analyte							
As	0.13	1.46	1.15	1.18	2-4	1.26	14
B	6.42	15.5	15.3	17.2	1	16.0	6.5
Be	0.012	1.32	1.28	1.35	1-2	1.32	2.7
Ce	<0.079	<0.077	<0.075	<0.072		<0.077	
Co	0.014	0.356	0.331	0.369	1-3	0.352	5.5
¹³³ Cs	0.014	4.96	4.90	5.40	1	5.09	5.4
Eu	<0.017	<0.016	<0.016	<0.015		<0.016	
¹²⁷ I	<0.013	2.42	2.49	2.73	2-5	2.55	6.4
Li	<0.064	0.323	0.329	0.367	1	0.340	7.0
Mo	<0.013	15.9	13.4	14.1	1-2	14.5	8.9
Pr	<0.018	<0.018	<0.017	<0.017		<0.018	
Rb	0.017	3.92	3.78	4.22	1	3.97	5.7
Sb	0.029	0.045	0.041	0.040	4-10	0.042	7.2
Se	<2.3	<2.3	<2.3	<2.3		<2.3	
Ta	<0.087	<0.087	<0.089	<0.089		<0.089	
Te	<0.17	<0.17	<0.16	<0.16		<0.17	
Th	<0.011	<0.011	<0.011	<0.011		<0.011	
Tl	<0.0055	0.0192	0.0168	0.0165	3-8	0.018	8.5
U	<0.14	58.9	52.9	54.1	3.5	55.3	5.7
V	<0.79	<0.77	<0.75	<0.72		<0.77	
W	<0.089	28.5	28.4	28.9	2-5	28.6	0.9
²⁴¹ AMU	<0.011	<0.011	<0.011	<0.011		<0.011	
²⁴² AMU	<0.010	<0.010	<0.010	<0.010		<0.010	
²⁴³ AMU	<0.0069	<0.0071	<0.0072	<0.0072		<0.0072	

Note: the last 3 listed elements, AMU-241, 242, and 243, were listed in this table with a concentration of $\mu\text{g/mL}$ as opposed to the radionuclide table in units of $\mu\text{Ci/mL}$. In order to determine the concentration in $\mu\text{Ci/mL}$, the mass would have to be assigned to a radioisotope and multiplied by the specific activity. Thus if AMU-241 is assigned to Am, then the activity becomes $<0.038 \mu\text{Ci/mL}$; if AMU-241 is assigned to Pu, then the activity becomes $<1.2 \mu\text{Ci/mL}$. The same concept is applicable to the other two masses.

(1) The error represents the error of each reported sample concentration. In some cases, the error was identical and a single error value is given. In other cases, the reported values had varying errors and the error range is given.

Table 3.4. 241-AP-101 Composite, IC Results

<i>Lab ID</i>	00-1701	00-1701D	00-1701T				Measured	Specification 7
<i>Sample ID</i>	sample	duplicate	triplicate	Average	RSD	Average	Mole analyte	Envelope A
<i>Units</i>	µg/mL	µg/mL	µg/mL	µg/mL	%	M	per mole Na	Limits
							Ratio	Ratio
Inorganic Anions ⁽¹⁾ , run date 4/23-24/00								
Fluoride ⁽²⁾	2,880	2,880	2,940	2,900	1.2	1.5E-1	2.7E-2	9.1E-2
Chloride	2,000	1,920	2,010	1,980	2.5	5.6E-2	9.9E-3	3.7E-2
Nitrite	42,200	41,000	42,400	41,900	1.8	9.1E-1	1.6E-1	3.8E-1
Nitrate	133,000	130,000	133,000	132,000	1.3	2.1E+0	3.8E-1	8.0E-1
Phosphate	1,040	1,000	1,020	1,020	2.0	1.1E-2	1.9E-3	3.8E-2
Sulfate	4,070	3,940	4,080	4,030	1.9	4.2E-2	7.5E-3	1.0E-2
Organic Anions ⁽³⁾ , run date 5/11-13/00								
Acetate ⁽⁴⁾	1,540	1,790	1,600	1,640	8.0			
Formate	1,130	1,260	1,200	1,200	5.4			
Oxalate	1,800	1,780	1,820	1,800	1.1			
Citrate	<890	<890	<890					
Additional analyte information from Inorganic Anion analysis								
Bromide	<500	<500	<500					

(1) Overall error for reported results is estimated to be within +/-15%, 2-σ.

(2) The fluoride results represents the summation of fluoride, acetate, and formate.

(3) Overall error for the reported results is estimated to be within +/-30%, 2-σ.

(4) The acetate value represents the summation of acetate and glycolate.

Table 3.5. 241-AP-101 Composite, Miscellaneous Analyte Results

										Specification 7 Envelope A Limits
<i>Lab ID</i>	PB-1701	00-1701	00-1701D	00-1701T					Measured	
<i>Sample ID</i>	prep blank	sample	duplicate	triplicate	Average	RSD	Average		Mole analyte	Mole analyte
<i>Units</i>	µg/mL	µg/mL	µg/mL	µg/mL	µg/mL	%	M	Run Date	per mole Na	per mole Na
Analyte ⁽¹⁾									Ratio	Ratio
Persulfate Method ⁽²⁾										
TIC		6,550	6,400	6,430	6,460	1.2	5.4E-1	4/13-14/00	9.58E-2	3.0E-1
TOC		1,860	1,980	2,030	1,960	4.5	1.6E-1	4/13-14/00	2.91E-2	5.0E-1
TC ⁽³⁾		8,410	8,390	8,460	8,420	0.4		4/13-14/00		
TC, Furnace Method ⁽²⁾		8,530	8,670	8,620	8,610	0.8		4/13-14/00		
Mercury	0.026	<0.025	<0.025	0.032			<2E-7	5/11/00	<3E-8	1.4E-5
Cyanide		5.81	5.56	5.73	5.70	2.2		5/11/00		
Ammonia ⁽⁴⁾		1.83	1.70	1.61	1.71	6.5		4/11/00		
Total hydroxide		42,300	41,100	40,500	41,300	2.2	2.4E+0	5/11/00		
<i>Units</i>	µg/L	µg/L	µg/L							
Total PCB	<1.4	<1.4	<1.4					8/14/00		
<i>Units</i>		g/mL	g/mL	g/mL	g/mL	%				
Density		1.307	1.309	1.308	1.308	0.076		4/5/00		

(1) Overall error for the reported results (except for ammonia) is estimated to be within +/-15%, 2-σ.

(2) Reporting units are µg C/mL solution.

(3) Total carbon is the sum of the TIC and TOC of the persulfate oxidation method.

(4) Error is estimated to be +/-20%, 2-σ.

4.0 Procedures, Quality Control, and Data Evaluation

A discussion of procedures, data quality, and quality control is provided below for each analytical method. The PNL-ALO-128 preparative blank spike and matrix spike recoveries are relevant to ICP and specifically included TCLP metals. However, two of the TCLP metals (As and Se) are included in the ICP-MS analysis. Other analytical methods, as well as ICP, generally relied on post digestion blank spikes and post digestion matrix spikes for use in data quality assessments. Analytical instrument calibration and calibration verification was performed in accordance with the SBMS Quality Assurance Program. This QA program is in compliance with HASQARD. Raw data including bench sheets, instrument printouts, data reduction, and calibration files are maintained or cross-referenced in the project files.

4.1 Metals Analysis by ICP—Tables 3.1 and 4.1

The PNL-ALO-128 acid extracted samples required 5, 10 and 50-fold dilutions in order to quantify all analytes of interest according to PNL-ALO-211. The detected analytes at or above the estimated quantitation level (EQL) are reported with an uncertainty of $\pm 15\%$ (2σ), and is equivalent to ten times the MDL. Values in brackets “[]” are less than the EQL, and have uncertainties greater than $\pm 15\%$. Non-detected analytes are reported as less than the MDL. As the MDL is approached, uncertainty increases to 100%.

Quality control for the ICP analysis consists of sample duplicates, process blanks, serial dilution, matrix spikes, laboratory control standards (LCS) or blank spikes, post spikes, verification check standards, and high calibration standards. Matrix spike and LCS recovery and precision quality control parameters were also defined by BNFL. These quality control parameters were evaluated in detail and are summarized below.

Replicates: All analytes of interest were recovered within the precision tolerance limit of $\leq 15\%$ relative standard deviation (RSD) except for P where the RSD was 16%.

Preparation blanks: All preparation blank analytes of interest were within tolerance limit of \leq EQL or $< 5\%$ of sample concentration in the prepared samples.

Serial dilution: Results from serial dilution were within tolerance limit of $\leq 10\%$ for all analytes tested after correcting for dilution.

Matrix spikes: All matrix-spiked analytes of interest were recovered within tolerance limits of 75% to 125% recoveries except silver, barium and lead. Silver recovery (about 35%), barium recovery (about 30%) and lead recovery (about 65%) were all low and may be related to the presence of sulfate and/or carbonate in the sample. Low silver recovery is probably due to the small amount of hydrochloric acid used during sample processing resulting in some silver chloride precipitation.

LCS: All LCS aliquots had a small amount of precipitate remaining after processing that required filtration. All analytes of interest were recovered within tolerance limit of 80% to 120% recoveries except silver. Low recovery of silver in the blank spike (about 69%) was probably due to the small amount of hydrochloric acid used during sample preparation resulting in some silver chloride precipitating from solution. The LCS for silver recovery was 69%, below QC Acceptance Criteria of 80%-120%.

Post-spiked samples: All analytes of interest were recovered within tolerance of 75% to 125% except silicon and palladium. Silicon recovery (147%) exceeded tolerance limit. Palladium recovery (53%) exceeded tolerance limit. All other analytes of interest above EQL were within tolerance. Palladium was found low in the group B spike. Single element palladium at 2.0 µg/ml measured at the beginning, middle and end of the run, were well within the tolerance limit. Palladium was not detected in the sample aliquots.

Verification check standards: Concentrations of all analytes were within the tolerance limit of $\pm 10\%$ accuracy in standards (except palladium). The calibration blank concentration was acceptable, less than two times the IDL. The palladium in the QC check standard was low (about 42% to 48%), however, a single element standard of palladium at 2.0 µg/ml measured at the beginning, middle and end of the ICP run was well within tolerance limit indicating acceptable palladium accuracy. Palladium was not detected in the sample aliquots.

High calibration standard check: Verification of the high-end calibration for all analytes measured was within tolerance limits of $\pm 5\%$ accuracy.

Detection limits: The reported MDLs for all analytes were less than the BNFL MRQs and in most cases, the EQLs were less than the MRQs (Ba, La, Rh, Ru, and Si EQLs were higher than the associated MRQs).

4.2 Total Uranium (KPA) Tables 3.1 and 4.1

Triplicate PNL-ALO-106 acid digested samples were measured for total uranium according to procedure PNNL-ALO-4014 using Kinetic Phosphorescence Analysis (KPA). The triplicate results showed excellent agreement with a RSD of 1%. A small amount of uranium was detected in the acid digestion process blank; however, the level was a factor of 10,000 lower than the uranium measured in the samples. A blank prepared at the time of the analysis did not detect any uranium. Uranium continuing calibration verification (CCV) standards analyzed before and after the samples agreed within 3% of the known values. A specific blank spike and a matrix spike were not run. The U(KPA) analysis results agreed well with the U(ICP-MS) analysis results.

4.3 Radioisotopic Analyses, Tables 3.2 and 4.2

Gamma energy analysis (GEA), pertechnetate, tritium, ^{14}C , and ^{79}Se analyses were performed on as-received samples, i.e., acid digestion of sub-samples was not performed. Other radioisotopic analyses were performed using the PNL-ALO-106 acid digested samples. Because a radioisotopic LCS is not available, post digestion spikes, tracers, and/or carriers were used, as appropriate, for monitoring process control.

Gamma Spectrometry

Triplicate 100-µL aliquots of the AP-101 original material were prepared into 2-mL geometries with deionized water (no acid digestion was applied). The small aliquot size was necessary to reduce the detector dead time attributed to the high ^{137}Cs activity. The samples were counted and analyzed directly by GEA according to procedure PNL-ALO-450. Because no sample manipulation was performed other than simple aliquoting and diluting, no preparation blank was prepared. In order to meet the required detection limits, the samples were counted for 14 hours each. Despite the longer counting time, the ^{241}Am MRQ was not met; this isotope however was quantified using radiochemical separations. Only ^{137}Cs , ^{134}Cs , and ^{60}Co could be identified in the gamma spectra. The triplicate concentrations of ^{137}Cs , ^{134}Cs , and ^{60}Co agreed to within $\pm 3\%$, well within the $<15\%$ RSD acceptance

criteria. The other requested analyte concentrations were below the minimum reportable quantities. Analysis for ^{135}Cs by GEA was not possible because this isotope has no γ -emissions.

Pertechnetate

The radiochemical ^{99}Tc determination was requested to measure only Tc in the +7 oxidation state (pertechnetate). To this end, all sample manipulations had to be non-oxidizing so as not to alter the original Tc oxidation state. Small aliquots from the as-received material (no digestion) were taken for analysis according to procedure PNL-ALO-432. A blank was prepared and run in parallel with the samples. This procedure normally requires the use of a sodium dichromate addition to oxidize the Tc to the +7 oxidation state. The sodium dichromate addition was omitted and the procedure was otherwise performed as written. The separated fractions were then counted according to RPG-CMC-408. One sample was also counted by gamma spectrometry (according to PNL-ALO-450) to verify the absence of ^{137}Cs . Pertechnetate was added to the blank spike and matrix spike and their recoveries were 99% and 84%, respectively, well within project requirements. The sample activity propagated 1- σ uncertainty was 4%. The RSD of the triplicate samples was 8%, again well within the project-required <15% RSD. The blank contained no measurable ^{99}Tc with a detection limit well below the MRQ.

Comparison of the pertechnetate values to the ICP-MS generated values show excellent agreement. This indicates the ^{99}Tc present in AP-101 is primarily in the +7 oxidation state.

Total Alpha

The total alpha activity was determined by direct-plating small aliquots of the acid-digested samples onto planchets according to RPG-CMC-4001. The samples were then counted on Ludlum detectors according to RPG-CMC-408. The sample activities resulted in a 9% RSD, well within the <15% RSD acceptance criteria. Alpha activity was not found in the preparation blank. The blank spike ^{239}Pu recovery was 99% and the matrix spike ^{239}Pu recovery was only 55%. The low matrix spike recovery is attributed to the solids loading on the planchet from the sample matrix salts that attenuate/absorb the alpha emissions. This is a physical problem with the sample as it is presented to the detector and does not indicate the analysis is out of control. What this does indicate is that the analytical method (which does not correct for solids loading) is probably biased low by as much as 45% for this matrix. A better indication of the total alpha activity is given by the summation of the alpha emitters ($^{239+240}\text{Pu}$, ^{238}Pu , ^{241}Am , $^{243+244}\text{Cm}$ and ^{242}Cm), where matrix effects have been eliminated through radiochemical separations. These alpha-emitter summations range from 18% to 39% higher than the total alpha activity found by direct plating. The RSD for the summation method is $\pm 1\%$.

Total Beta

The total beta activity was determined by directly plating small aliquots of the acid-digested samples onto planchets according to RPG-CMC-4001. The samples were then counted on a low-background alpha/beta gas-flow proportional counter according to RPG-CMC-408. The detectors were calibrated for beta activity relative to $^{90}\text{Sr}/^{90}\text{Y}$. The beta energy of ^{137}Cs is similar to that of ^{90}Sr and will have a counting efficiency similar to that of pure ^{90}Sr . The total beta analyses compared to the ^{137}Cs determinations shows that virtually all of the beta activity is due to ^{137}Cs as the two activity concentrations are in excellent agreement. The 5% RSD is within the experimental error. The blank spike and matrix spike showed excellent $^{90}\text{Sr}/^{90}\text{Y}$ recoveries at 100% and 103%, respectively.

Plutonium, Americium, and Curium

Analyses of Pu, Am, and Cm were conducted on the acid-digested samples. The Pu and Am/Cm separations were performed according to PNL-ALO-417; the separated fractions were precipitation plated according to PNL-ALO-496; and the samples were counted by alpha spectrometry according to

PNL-ALO-422. Plutonium recovery was traced with ^{242}Pu . The curium chemistry is known to follow the americium and both these isotopes were traced with ^{243}Am . Plutonium radiochemical yields were excellent at >90%. Radiochemical yields for Am ranged from 70% to 86%. In both cases, ample counts were obtained to define analyte and tracer activities. Neither Pu, Am nor Cm were detected in the preparation blank. The blank spike and matrix spike for Pu resulted in 98% and 101% yield-corrected recovery. This indicates the chemistry and analyses were not biased. The blank spike and matrix spike for the Am (and Cm) fractions resulted in 94% and 91% yield-corrected recovery. The $^{239+240}\text{Pu}$ RSD was 2% and the ^{238}Pu RSD was 5%. The ^{241}Am RSD was 2% and the $^{243+244}\text{Cm}$ RSD was 29%. This latter high RSD reflects the much higher uncertainty associated with the analytical results, which were approximately 5 orders of magnitude less than the requested MRQ.

Strontium-90

The Sr separation was performed on the acid-digested samples according to PNL-ALO-476 and radiochemical yields were traced with ^{85}Sr . The separated fractions were then beta-counted according to RPG-CMC-408 and gamma counted according to PNL-ALO-450 (for ^{85}Sr determination and ^{137}Cs impurity assessment). Two of the separated fractions contained a small amount of ^{137}Cs and a correction to the beta count rate was applied for these samples. Strontium-90 was not found in the preparation blank. The RSD was 0.3%, indicative of excellent precision. The LCS and matrix spike recoveries were 92% and 99%, respectively, indicating good accuracy.

Tritium

Aliquots of AP-101 composite were diluted in water and distilled for ^3H determination according to procedure PNL-ALO-418. Two serial distillations were required to isolate tritium from ^{137}Cs . Tritium was then measured using liquid scintillation counting according to procedure PNL-ALO-474. The first distillation showed the presence of higher-energy beta activity, most likely due to ^{137}Cs . A second distillation succeeded in removing most of this contamination; however, two of the triplicate samples required subtraction of weak beta contamination using the ratio of the counts in the tritium energy region to the counts at higher energies, as determined from the first counting results. The triplicate results showed good agreement with a RSD of 5%. A blank spike showed good recovery at 96%. No tritium was detected in a blank processed with the samples.

Selenium-79

Direct AP-101 composite sub-samples were analyzed in triplicate for ^{79}Se following procedure PNL-ALO-440. Each sample was spiked with 20 mg Se carrier in solution for yield correction. The samples were passed through mixed-bed anion and cation exchange resins that removed most radiochemical interferences. Selenium was distilled as selenium bromide and then reduced to elemental form. The chemical yield was determined gravimetrically by weighing the recovered elemental selenium. The selenium was then dissolved and the ^{79}Se activity was determined by liquid scintillation counting according to procedure PNL-ALO-474. The liquid scintillation spectra did not show clear evidence for a ^{79}Se peak and there appeared to be some weak beta contamination evident in the higher energy region of the beta spectrum. Hence, the results probably have a slight high bias. The measured ^{79}Se activity (averaging $5.8\text{E-}5 \mu\text{Ci/ml}$) was below the requested MRQ of $9.0\text{E-}5 \mu\text{Ci/ml}$ and only slightly above the nominal MDL of $2.0\text{E-}5 \mu\text{Ci/ml}$. The sample RSD of 18% exceeded the RSD acceptance criterion of <15%; however, measurement uncertainties were as high as 11% (1- σ) and the sample activities were only a factor of three higher than the MDL. A ^{79}Se standard was not available, thus ^{14}C (similar beta endpoint energy as ^{79}Se) was used to calibrate the liquid scintillation detector. For the same reason a blank spike and a sample spike could not be run. The sample carrier recoveries were generally low at 27% to 63% and the blank recovery was 68%. All sample activities were corrected for radiochemical yield. The process blank did not show any evidence of contamination.

Carbon-14

Direct samples of the AP-101 composite were analyzed in triplicate for ^{14}C following procedure PNL-ALO-482. The samples were combusted in a Coulometrics Carbon Analyzer Furnace causing oxidation of all carbon species present to CO_2 . A natural carbon compound was added as a carrier and all of the CO_2 released was collected in a sodium hydroxide trap. An aliquot of the trap solution was then counted by liquid scintillation counting according to procedure PNL-ALO-474. The triplicate results showed good agreement with a RSD of 5%. The blank spike and sample spike showed good recoveries at 97% and 96%, respectively. Carbon-14 was not detected in the blank.

4.4 Inductively-Coupled Plasma—Mass Spectrometry, Tables 3.3. and 4.3

Splits from the acid extraction (PNL-ALO-128) were submitted for ICP-MS analysis according to procedure PNL-ALO-280, Rev. 1. The sample splits were from the same processed solutions as were delivered for ICP analysis. A preparative blank and TCLP-spiked blank and sample were also submitted for ICP-MS analysis (As and Se were the relevant spiked analytes).

Values for the following isotopes were obtained using responses from related isotopes: ^{126}Sn (obtained from ^{118}Sn), ^{231}Pa (obtained from ^{232}Th), and ^{240}Pu , ^{241}AMU , ^{242}AMU , ^{243}AMU (obtained from ^{239}Pu). Because the concentrations of these isotopes were determined indirectly, ^{126}Sn , ^{231}Pa , ^{240}Pu , ^{241}AMU , ^{242}AMU , and ^{243}AMU results should be considered semi-quantitative.

In general, the results for the quality control samples (i.e., calibration verification standards, duplicates, blank spike, sample spike, and post digestion sample spikes) were well within acceptance criteria. One of the CCV results for tellurium and tantalum and the spike recovery for thallium, were outside the acceptance window (<20% for the CCV and <30% for the Spike). Tellurium and tantalum did have another CCV result that was acceptable. The CCV results for thallium were acceptable. The RSDs for all detected analytes were <15%. The MDL for ^{231}Pa was slightly above the MRQ. The preparation blank for As and B represented 10% and 40% of the measured sample concentrations, higher than the 5% acceptance criteria defined in the QA plan.

The ^{99}Tc values reported assume that the Ru present is exclusively fission-product Ru, and therefore does not have an isotope at mass to charge ratio (m/z) 99; i.e., everything observed at m/z 99 is due to ^{99}Tc . From the appearance of the Ru isotopic abundance, this appears to be a reasonable assumption; the isotopic fingerprint exhibited is not natural. Good agreement between the total ^{99}Tc and the pertechnetate (radiochemical determination) values were obtained.

4.5 Anion Analysis, Tables 3.4 and 4.4

Anion analysis was conducted according to two different methods in an effort to achieve the desired specificity. One method, PNL-ALO-212, Determination of Inorganic Anions by Ion Chromatography, is optimized for determination of inorganic anions. The other method, AOAM-1, Method for the Analysis and Quantification of Organic Acids in Simulated and Actual Hanford Tank Wastes by Ion Chromatography, is optimized for the determination of organic acids. Both methods were used to evaluate the anionic analytes of interest on unprocessed AP-101 composite sub-samples. Oxalate concentration was determined by the inorganic anion method, however the reliability of the organic anion method is considered to provide superior oxalate results.

Inorganic Anions

The AP-101 composite sub-samples were diluted 2000 to 5000 fold at the IC workstation to ensure that all anions reported were measured within the calibration range. All client-defined MRQs were met at the dilutions analyzed. From recoveries demonstrated on some of the verification standards, the AP-101 sample matrix had an adverse effect on the IC column and some reported results may be 10% to 20% low. By further diluting the sample, this effect could be minimized, but at the expense of meeting the MRQs (particularly phosphate and sulfate).

For the IC column and parameters used, the IC system cannot separate fluoride, acetate, and formate; the IC system quantifies and reports all as fluoride. Acetate (and/or glycolate) and formate were quantified in the organic anion analysis, the sum of these nearly equaling the “fluoride” concentration. An upper bound to the fluoride concentration can be calculated by subtracting the acetate and formate sum from the reported fluoride concentration providing 50+/- 960µg F/mL or <1000 µg F/mL.

Duplicates: The RSD was <3% for all anions, well within the <15% RSD acceptance criterion.

Matrix Spike: The matrix spike recoveries for all anions, except nitrate, were within the spiking acceptance criteria of 75% to 125%. The high nitrate concentration of the sample relative to the nitrate spiking level (i.e., sample concentration was 10 times greater than the spike concentration) significantly impacts the ability to measure the nitrate spike. No attempt was made to spike the samples at an additional spiking level. A 10,000x dilution provided a result of 130,000µg/mL nitrate, in good agreement with the 2000x dilution. This indicates the matrix effects on nitrate determination are negligible.

Blank Spike: The blank spike recoveries for all anions, except nitrate, were within the Laboratory Control Samples (LCS) acceptance criteria of 80% to 120%. The nitrate recovery was slightly low at 78%. It should be noted that the blank spike nitrate recovery was not consistent with other control standards analyzed during the analysis run (e.g., Verification Check Standards).

System Blank/Processing Blanks: A dozen system blanks were processed during the analysis of the samples. No anions were detected in the system blanks above the method detection limit.

Quality Control Calibration Verification Check Standards: Over twenty mid-range verification standards were analyzed throughout the analysis runs. Numerous verification standards analyzed just after the analysis of some of the AP-101 samples demonstrated low recoveries (i.e., recoveries ranging from 80% to 90%). Failure of the verification standard was only observed in those standards analyzed just after the AP-101 samples that were prepared with the least dilution. Numerous reruns were performed alternating the AP-101 (at various dilutions) and the verification standard to obtain valid data.

Organic Anions

Sample AP-101 was diluted 2000-fold to give acceptable total ion loading on the column. The reported acetate concentration represents the summation of acetate and glycolate, since both ions co-elute under the sample analysis conditions. From tank waste and solubility studies, the acetate and glycolate concentrations are tank waste type dependent. That is, for one tank waste the component may be entirely acetate; for another tank with a different fill history, the component may be primarily glycolate, or a combination of both analytes (Sharma et al. 1998, Camaioni et al. 1998, Barney 1996, Ashby et al. 1994).

The RSD of the triplicate values were well within the <15% acceptance criteria. The method detection limits are given in Table 4.4. The oxalate MDL was below the client-directed MRQ; other analyte MRQs were undefined.

Matrix spiking was done in triplicate at a level approximately 1.5 times the level of analyte in the final diluted samples. This level of spiking is at a challenging level for accurate quantitation; the reported levels are near the lowest calibration point for each analyte. The acetate matrix spike recoveries were 175%, 91%, and 105%, averaging 124%. The formate matrix spike recoveries were 71%, 104% and 79%, averaging 84%. The oxalate matrix spike recoveries were 122%, 92%, and 129%, averaging 114%. The value for acetate in the matrix spike sample should be considered suspect, as the peak quantitation had to be done by estimation (since the peak size was so small). The other data represent quantitation that was extracted directly from electronic integration data. Overall, the average data for MS and MSD were within acceptable limits. The LCS consisted of a mixture of formate and oxalate and resulted in 89.3 and 103.8% recoveries, respectively.

4.6 Miscellaneous Analytes, Tables 3.5 and 4.5

Inorganic and Organic Carbon

The AP-101 composite analysis was performed by the hot persulfate wet oxidation method according to PNL-ALO-381 and by the furnace oxidation method according to PNL-ALO-380. The hot persulfate method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95°C for TOC, all on the same sample, with TC being the sum of the TIC and TOC. The furnace oxidation method determines TC by oxidizing all forms of carbon (i.e., inorganic and organic) in oxygen at 1000 °C. Although the ASR requested TOC and TIC by the furnace method, the method is considered reliable only for TC. For the sample matrix analyzed, the furnace method and hot persulfate method should provide equivalent TC results; this was confirmed, with the average hot persulfate TC being 8420 µg/ml and the furnace TC being 8610 µg/ml, an RPD of about 2%.

Per procedures, all sample results were corrected for average percent recovery of system calibration standards and were also corrected for contribution from the blank. Precision and bias were estimated to be $\pm 15\%$ (2σ).

The TIC standard is calcium carbonate and TOC/TC standard is α -Glucose. The standard materials were used in solid form for system calibration check standards as well as matrix spikes. The QC for the methods involves calibration blanks, system calibration standards, sample duplicates, and one matrix spike per matrix type.

Calibration Standards: The QC system calibration check standards were all within acceptance criteria of 90% to 110%, with the average recovery being about 99% for TIC and 99% for TOC over the two days that the hot persulfate analyses were performed and about 100% for TC for the furnace analyses.

Calibration Blanks: The calibration blanks run at the beginning, middle, and end of the analysis run were acceptable and the standard deviations for the TIC and TOC blanks were near the historical pooled standard deviation used to establish the method detection limits. On the May 12th run, the TOC blanks were about 2 to 3 times higher than normal, but were quite consistent. Because the blank results were subtracted from the sample results, the high blanks should have no effect on the reported data within the reported uncertainty.

Replicates: All RSDs were less than 5%, indicating good precision between all measurements. The RSDs met the acceptance criterion of <15%.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The matrix spike for both the hot persulfate and furnace method demonstrated recoveries of about 96% to 99% for TIC, TOC, and TC; well within the acceptance criteria of 75% to 125% recovery.

Laboratory Control Sample: No LCS was included in the carbon analysis procedure.

Mercury

The samples were analyzed by cold vapor atomic absorption spectrophotometry for inorganic mercury according to PNNL-ALO-201. Four aliquots of 0.2 ml including one for matrix spike, was processed and diluted to a final volume of 25 ml per procedure PNNL-ALO-131; no additional dilution was performed. Concentration of all sample aliquots measured were near instrument detection limit and several times lower than MRQ (1.5 µg/ml). The low calibration standard was defined as the instrument detection limit (DL) for the reported results and assumes non-complex aqueous matrices. Routine precision and bias is typically $\pm 15\%$ (2- σ) or better for non-complex aqueous samples that are free of interference.

Following are results of quality control checks performed during Hg analyses. In general, quality control checks met the requirements of the governing QA Plan.

Working Blank Spike/Process Blank Spike: The process blank spike recovery was 100%, well within the acceptance criteria of 80% to 120%.

Matrix Spiked Sample: A matrix spike was prepared for the samples submitted under this ASR. Recovery of the matrix spike was 97%, well within the acceptance criteria of 75% to 125%.

Duplicate/replicates: The RSD was not calculated since all replicate results were less than 5 x IDL.

Laboratory Control Sample (liquid): Sample recovery of mercury in SRM-1641d (certified by NIST to contain 1.60 ± 0.018 µg/ml) was recovered within acceptance criteria of 75% to 125%.

System Blank/Processing Blanks: A system blank was processed during the analysis of the sample. The concentration measured was within about two times detection limit or less. Samples were about the same concentration as the process blank.

Quality Control Calibration Verification Check Standards: Six mid-range verification standards were analyzed throughout the analysis run. All were within the acceptance criteria of 80% to 120% recovery for the verification standard.

Cyanide

The AP-101 composite samples were distilled according to PNL-ALO-287 with the addition of sulfamic acid to minimize interference from high nitrates present in the sample. The distillates were analyzed for CN concentration according to PNL-ALO-289. The reporting limits were estimated to be approximately 0.25 µg CN/ml based on the sample quantity distilled. No quality control or other measurement problems were encountered.

An independent mid-range calibration check solution run at the beginning, middle, and end of the analysis batch gave an average recovery of 100%. These calibration check standards ranged from 99% recovery to 100% recovery, which is within the 85% to 115% acceptance criteria of the governing QA plan.

The AP-101 composite was analyzed in triplicate. The RPD between the sample and duplicate was 4.4% and the RSD for the triplicates was 3.1%. The measurement precision met the quality control criterion of <15% RSD. The LCS (a distilled blank spike) recovery was 101%, well within the quality control acceptance criteria. The spike recovery for the spiked AP-101 composite was 95%, well within the acceptance criteria of 75% to 125%.

Ammonia

Ammonia was measured in triplicate sample aliquots of AP-101 composite using an ion selective electrode according to procedure PNL-ALO-226. The ammonia probe was calibrated using five standards spanning ammonia concentrations from 1.0E-2 to 1.0E-6 M. The method of standard additions was used to determine the ammonia concentrations by first taking a direct reading and then adding a known standard to each sample. The triplicate concentrations were in good agreement with a RSD of 6%. The method detection limit was estimated at 0.2 µg/ml, well below the requested MRQ value of 140 µg/ml.

Hydroxide

A sample of the AP-101 composite was analyzed in triplicate for the free hydroxide content following procedure PNL-ALO-228. Direct sample aliquots were analyzed using a Brinkman 636 Auto-Titrator. A 0.1186 N NaOH solution was prepared for use as a standard and sample spike and the titrant was a 0.2040 M HCl prepared solution. Triplicate determinations resulted in ±3% RSD. The standard recoveries averaged 100% and the sample spike recovery was 88%. Hydroxide was not detected in the reagent blank, demonstrating an MDL of 0.02M OH or 340 µg OH/mL.

PCB

Sample handling processes were inconsistent with a procedure that would be employed for low concentration PCB analysis in that sample vessels were not rinsed with a PCB miscible solvent following sample transfer. The rinse is employed to remove adsorbed PCBs from container walls, ensuring complete analyte transfer. However, the client-requested MRQ of 3300 µg/kg (4300 µg/L) is much greater than a concentration of PCBs where adsorption issues would be of concern such as nanogram per milliliter quantities. Thus any potential loss of analyte would be of little consequence. However, if PCBs were present in the sample, the actual detection limit may have been affected and is potentially higher than reported. Despite the potential bias, the actual method detection limit would be substantially below the MRQ, particularly since there is more than three orders of magnitude between the reported detection limit and MRQ.

Because limited sample was available, the quantity of sample used for spiking was approximately half of that used for unspiked sample analysis. The LCS consisted of a spiked blank at a volume similar to that used for the samples (100 mL). The LCS recovery is less than the 80-120% recommended by USEPA SW-846 method 8082. However, the spiking level employed is considerably lower than the levels used in the SW-846 method by as much as 25 times. The lower spiking level used is more consistent with up-to-date regulatory expectations. The recovery does meet the general acceptance criteria of 70-130% referred to in USEPA method 8000, which would be applied since the spiking level used falls outside of the range applied in USEPA method 8082.

While no specific surrogate acceptance criteria have been developed for tank matrices, the recoveries are all within recommended initial criteria of 20-150%, and meet USEPA CLP SOW OLM01.8 advisory criteria of 60-150% for water.

Density

The density of the AP-101 composite was calculated using the net sample mass in a 10-mL volumetric flask at ambient temperature. The RSD of the measurements was 0.08%.

Table 4.1. 241-AP-101 Composite, ICP Metals QC Results

Table 11.1. EPA-IR-101 Composite, POP Metals QC Results								
Lab ID				BNFL			Matrix	Post
Sample ID	Average	RSD	MDL	MRQ	LCS	Spike	Matrix Spike (A)	Matrix Spike (B)
Units	µg/mL	%	µg/mL	µg/mL	% Recovery	% Recovery	% Recovery	% Recovery
Acceptance Criteria		<15% ⁽³⁾			80%-125%	75%-125%		
Analyte ⁽¹⁾								
Ag	<0.63		0.63	17	69	35	96	
Al	6,940	5.5	1.5	75			113	
Ba	[0.33]	3.0	0.25	2.3	87	30	96	
Bi	<2.5		2.5	170			95	
Ca	[7.7]	2.0	6.3	150			97	
Cd	[2.0]	2.9	0.38	7.5	92	87	97	
Cr	143	8.9	0.51	15	93	94	103	
Cu	[1.6]	7.4	0.63	17			97	
Fe	[2.5]	14	0.63	75			97	
K	31,200	2.1	51	750			115	
La	<1.3		1.3	2.3				100
Mg	<2.5		2.5	170			102	
Mn	<1.3		1.3	17			99	
Na	129,000	2.9	3.8	170				
Nd	<2.5		2.5	170				101
Ni	7.9	7.6	0.76	30	88	85	99	
P	314	16	2.5	330			98	
Pb	[15]	0.0	2.5	300	101	65	107	
Pd	<19		19	390				53
Rh	<7.6		7.6	18				95
Ru	<28		28	36				96
Si	137	4.0	13	90			147	
Sr	<0.38		0.38	17			97	
Ti	<0.63		0.63	17			95	
U	[62]	9.7	51	780				104
U(KPA) ⁽²⁾	51.1	1.4	0.0002	780				
U(ICP-MS) ⁽²⁾	55.3	5.7	0.02	NP			115	
Zn	[5.6]	3.7	1.3	17	89	92	96	
Zr	[1.4]	4.2	1.3	17			100	
Additional analyte information								
As*	<6.3				95	[97]	107	
Be*	[1.0]	5.6					97	
B*	15	2.6					97	
Co*	<1.3						97	
Li*	<0.76						95	
Mo*	[11]	11					97	
Se*	<6.3				95	[80]	104	
Sn	<38							93
Tl*	<13				92	[91]	97	
V*	<1.3						97	
W*	<51							95
Y	<1.3						99	

Shaded areas highlight non-compliances with BNFL acceptance criteria, see report for discussion.

(1) Overall error for reported results is estimated to be within +/-15% 2-σ; however results in brackets "[]" are less than the estimated quantitation level (i.e., 10-times MDL) and error is anticipated to exceed +/-15%.

(2) U (KPA) indicates U determination by kinetic phosphorescence; U(ICP-MS) indicates U determination by ICP-MS. QC parameters for these techniques were not specified.

(3) RSD acceptance criteria for Na concentration is <3.5%.

*Analytes on ICP-MS Specification 7 list.

Table 4.2. 241-AP-101 Composite, Radionuclide QC Results

	00-1701				BNFL	Blank	Blank spike		MS
	Average	% Err	RSD ⁽³⁾	MDL	MRQ	Spike	Acceptance	Matrix	Acceptance
Units	μCi/mL	1-σ	%	μCi/mL	μCi/mL	% Recovery	Criteria	Spike	Criteria
							% Recovery	% Recovery	% Recovery
Radiochemical Analytes									
³ H	4.83E-3	3	4.7	2.0E-4	2.1E-2	96	80-120		N/A ⁽⁵⁾
¹⁴ C	2.63E-4	8	4.7	4.0E-5	7.2E-4	97	80-120	96	75-125
⁶⁰ Co (GEA) ⁽¹⁾	3.29E-3	7	3.0		2.1E-3		NP ⁽⁴⁾		N/A
⁷⁹ Se	5.82E-5	5	18	3.0E-6	9.0E-5		NP		N/A
⁹⁰ Sr	8.74E-2	3	0.3	2.0E-4	3.0E-2	92	75-125	99	N/A
⁹⁹ Tc (+7)	4.72E-2	4	8.1	2.0E-5	1.5E-3	99	80-120	84	70-130
¹⁰⁶ Ru/Rh (GEA) ⁽¹⁾	<9E-2			9.0E-2			NP		N/A
¹²⁵ Sb (GEA) ⁽¹⁾	<6E-2			6.0E-2	1.7E+0		to be obtained		N/A
¹³⁴ Cs (GEA) ⁽¹⁾	4.73E-2	3	2.1		3.9E-2		NP		N/A
¹³⁷ Cs (GEA) ⁽¹⁾	1.44E+2	2	0.4		3.9E-1		NP		N/A
¹⁵² Eu (GEA) ⁽¹⁾	<2E-3			2.0E-3			NP		N/A
¹⁵⁴ Eu (GEA) ⁽¹⁾	<2E-3			2.0E-3	2.0E-2		NP		N/A
¹⁵⁵ Eu (GEA) ⁽¹⁾	<4E-2			4.0E-2	9.0E-2		NP		N/A
²³⁸ Pu	1.47E-5	6	5.2	2.0E-7	1.0E-2		NP		N/A
²³⁹⁺²⁴⁰ Pu	1.26E-4	4	1.6	2.0E-7	1.0E-2	98	NP	101	N/A
²⁴¹ Am (GEA) ⁽¹⁾	<4E-2			4.0E-2	1.0E-2		NP		N/A
²⁴¹ Am	1.69E-4	5	2.4	5.0E-7	3.0E-2	94	NP	91	N/A
²⁴² Cm	<2E-7			2.0E-7	1.5E-1		NP		N/A
²⁴³⁺²⁴⁴ Cm	1.03E-6	21	29	8.0E-8	1.5E-1		NP		N/A
Total alpha	2.47E-4	14	9.1	7.0E-5	2.3E-1	99	70-130	55	70-130
Alpha sum ⁽²⁾	3.11E-4		0.7						
Total beta	1.47E+2	4	4.8	2.0E-1		100	70-130	103	70-130
ICP-MS Analytes									
⁹⁹ Tc (total)	4.90E-2	1-4	4.3	2.6E-4	1.5E-3		80-120	101	70-130
¹²⁶ Sn	<9E-4			9.0E-4	6.0E-3		80-120		70-130
¹²⁹ I	8.05E-5	3-7	7.1	6.7E-6	1.8E-5		NP	99	N/A
²³¹ Pa	<1E-4			1.1E-4	7.9E-5		developed by lab		developed by lab
²³³ U	5.30E-5	7-11	8.2	8.8E-6	4.2E-4		90-110		75-125
²³⁴ U	2.40E-5	12-21	12.5	5.7E-6	1.2E-4		90-110		75-125
²³⁵ U	1.01E-6	0.3	4.2	6.8E-9	4.5E-8		90-110		75-125
²³⁶ U	2.13E-6	2-6	4.2	5.8E-8	1.4E-6		90-110		75-125
²³⁸ U	1.84E-5	0.5	5.5	4.7E-8	7.2E-8		80-120		70-130
²³⁷ Np	<6E-6			5.4E-6	3.9E-5		90-110	119	75-125
²³⁹ Pu	<2E-3			1.2E-3	3.0E-2		not provided	110	not provided
²⁴⁰ Pu	<3E-3			2.4E-3	3.0E-2		not provided		not provided

Shaded areas highlight non-compliances with BNFL acceptance criteria, see report for discussion.

(1) Samples were run directly by GEA and did not require a preparation blank.

(2) Alpha sum refers to the summation of ²³⁸Pu, ²³⁹⁺²⁴⁰Pu, ²⁴¹Am, and ²⁴³⁺²⁴⁴Cm alpha activities.

(3) In all cases the QC acceptance criteria for the RSD is <15%.

(4) NP: not performed, as stated in Quality Control Parameters for Liquid Analysis

(5) N/A: not applicable, as stated in Quality Control Parameters for Liquid Analysis

Table 4.3. 241-AP-101 Composite, ICP-MS Stable Element QC Results

	Average	RSD	MDL	BNFL		MS	Post Matrix Spike
<i>Units</i>	$\mu\text{g/mL}$	%	$\mu\text{g/mL}$	$\mu\text{g/mL}$	% recovered	% recovered	% recovered
Acceptance Criteria		<15%			80%-120%	70%-130%	70%-130%
Analyte							
As	1.26	14	0.063	2.3	117	120	109
B	16.0	6.5	0.16	2.3			105
Be	1.32	2.7	0.004	2.3			105
Ce	<0.077		0.077	2.3			117
Co	0.352	5.5	0.012	2.3			108
$^{133}\text{Cs}^{(1)}$	5.09	5.4	0.004	1.5			108
$\text{Eu}^{(1)}$	<0.016		0.016	60			115
$^{127}\text{I}^{(1)}$	2.55	6.4	0.037	TBD ⁽²⁾			94
Li	0.34	7.0	0.063	2.3			123
Mo	14.5	8.9	0.013	2.3			105
Pr	<0.018		0.018	2.3			118
Rb	3.97	5.7	0.011	2.3			106
Sb	0.042	7.2	0.014	2.3			94
Se	<2.3		2.3	2.3	102	118	
Ta	<0.089		0.089	2.3			89
Te	<0.17		0.17	2.3			73
Th	<0.011		0.011	2.3			122
Tl	0.0180	8.5	0.011	2.3			139
V	<0.77		0.77	2.3			110
W	28.6	0.9	0.23	2.3			124
$^{241}\text{AMU}^{(1)}$	<0.011		0.011				
$^{242}\text{AMU}^{(1)}$	<0.010		0.010				
$^{243}\text{AMU}^{(1)}$	<0.0072		0.0072				

Shaded area highlights non-compliance with BNFL acceptance criteria, see report for discussion.

(1) QC acceptance criteria for LCS and matrix spike recoveries and precision were not specified.

(2) To be determined by method

Table 4.4. 241-AP-101 Composite, IC QC Results

	Average	RSD	MDL	BNFL MRQ	LCS	Matrix Spike
<i>Units</i>	$\mu\text{g/mL}$	%	$\mu\text{g/mL}$	$\mu\text{g/mL}$	% recov.	% recov.
<i>Acceptance Criteria</i>		<15%			80%-120%	75%-125%
Inorganic Anions⁽¹⁾						
Fluoride ⁽²⁾	2,900	1.2	1.3E+2	1.5E+2	110	114
Chloride	1,980	2.5	1.3E+2	3.0E+2	107	111
Nitrite	41,900	1.8	2.5E+2	2.3E+3	105	119
Nitrate	132,000	1.3	2.5E+2	3.0E+3	78	128
Phosphate	1,020	2.0	2.5E+2	2.3E+3	104	119
Sulfate	4,030	1.9	2.5E+2	2.3E+3	106	108
Organic Anions⁽³⁾						
Acetate ⁽⁴⁾	1,640	8.0	5.5E+2	TBD ⁽⁵⁾		124 ⁽⁶⁾
Formate	1,200	5.2	4.5E+2	TBD ⁽⁵⁾	89.3	84 ⁽⁶⁾
Oxalate	1,800	1.0	8.9E+2	1.8E+3	103.8	114 ⁽⁶⁾
Citrate	<890		8.9E+2	TBD ⁽⁵⁾		
Additional analyte information						
Bromide	<500				108	108

Shaded areas highlight non-compliances with BNFL acceptance criteria, see report for discussion.

(1) Overall error for reported results is estimated to be within +/-15%, 2- σ .

(2) The fluoride results represents the summation of fluoride, acetate, and formate concentrations as these cannot be resolved on the IC column.

(3) Overall error for reported results is estimated to be within +/-30% at 2- σ .

(4) Acetate concentration represents the summation of acetate and glycolate concentrations.

(5) To be determined by the method

(6) Based on the average of 3 matrix spike recoveries.

Table 4.5. 241-AP-101 Composite, Miscellaneous Analyte QC Results

	Average	RSD	MDL	BNFL MRQ	Blank Spike	Matrix Spike
<i>Units</i>	$\mu\text{g/mL}$	%	$\mu\text{g/mL}$	$\mu\text{g/mL}$	% Recovery	% Recovery
<i>Acceptance Criteria</i>		<15			80-120	75-125
Analyte						
Persulfate Method ⁽¹⁾						
TIC	6,460	1.2	4.0E+1	1.5E+2		98.5
TOC	1,960	4.5	8.0E+1	1.5E+3		96.3
TC	8,420	0.4	8.0E+1			97.5
TC, Furnace Method	8,610	0.8	1.7E+2			97.5
Mercury	<0.032		2.5E-2	1.5E+0	100	97
Cyanide	5.70	2.2	2.5E-1	4.5E+0	101	95
Ammonia	1.71	6.5	2.0E-1	1.4E+2		
Total hydroxide	41,300	4.2	3.4E+2	7.5E+4	100	88
<i>Units</i>	$\mu\text{g/L}$	%	$\mu\text{g/L}$	$\mu\text{g/L}^{(3)}$	%	%
Total PCB	<1.4		1.4	4300	71.8	101 ⁽⁴⁾
<i>Units</i>	g/mL	%	g/mL	g/mL		
Density ⁽²⁾	1.308	0.076		0.9		

(1) Reporting units are $\mu\text{gC/mL}$ solution.

(2) Acceptance criteria for precision was not specified.

(3) MRQ of $3300\mu\text{g/kg}$ corrected for density.

(4) Average of MS (106%) and MSD (96.9%) recoveries.

5.0 References

Sharma, A. K., S. A. Clauss, G. M. Mong, K. L. Wahl, J. A. Campbell. 1998. *Analysis and Quantification of Organic Acids in Simulated Hanford Tank Waste and Hanford Tank Waste*. Journal of Chromatography, 805, 101-107.

Camaioni, D. M., W. D. Samuels, J. C. Linehan, S. A. Clauss, A. K. Sharma, K. L. Wahl, J. A. Campbell. 1996. Organic Tanks Safety Program, FY 96 Waste Aging Studies, PNNL-11312, Pacific Northwest National Laboratory, Richland, Washington.

Ashby, E. C. et al. 1994. *Synthetic Waste Chemical Mechanism Studies*, WHC-EP-0823, Westinghouse Hanford Company, Richland, Washington.

Barney, G. L. 1996. *Solubilities of Significant Organic Compounds in HLW Tank Supernate Solutions-FY 1996 Progress Report*, WHC-EP-0899, Westinghouse Hanford Company, Richland, Washington.

APPENDIX A

Appendix A: Tank 241-AP-101 Grab Samples

APPENDIX B

Appendix B: Test Instructions

APPENDIX C

Appendix C: Analytical Reports

Polychlorinated Biphenyls/Pesticides Analysis

Introduction

Analysis of 241-AP-101 liquid composite samples for Polychlorinated Biphenyls (PCBs) was performed by the AOAM group at PNNL. 100 mL sample aliquots were prepared using liquid-liquid extractions in the SAL. This procedure is outlined in the test instructions found in appendix B, of the technical report PNWD-3046 *Inorganic, Radioisotopic, and Organic Analysis of 241-AP-101 Tank Waste*, June 2000. The resulting methylene chloride residues were transferred under COC to the laboratory for concentration, cleanup, and analysis for PCBs. The analysis was performed using gas chromatography/electron capture detection (GC/ECD) and confirmation by mass spectrometry if necessary. The PCB analysis was not included in the previously referenced report pending authorization to proceed and is presented separately as this addendum.

Sample Preparation

300 mL of composite 241-AP-101 sample material was available for PCB analysis. Prior sample handling indicated the composite samples were combined from a number of containers. For ascertaining low concentrations of PCBs, each of these and the container used to perform the composite, should be rinsed with a PCB miscible solvent to obtain an accurate sample representation. The rinsing process is performed because the solubilities of PCBs are quite low in aqueous samples and are subject to “plating out” on the surfaces of sample containers. The solubility of PCBs is lower in high salt solutions such as tank supernate material. However, this procedure was not performed for this analysis.

Extractions for PCBs were performed according to the test plan, BNFL-TP-29953-089, *Organic Extraction of AP-101 for PCBs*. Duplicate 100 mL and 50 mL aliquots of the liquid samples were obtained for extraction. For the sample and sample duplicate preparations, 100 mL each was used. For the spike and spike duplicate samples, 50 mL aliquots were used for each preparation. 0.04 ug of each surrogate compound was added to all samples. 0.20 ug of Aroclor 1254 was added to the spike samples. Each of the samples were liquid-liquid extracted in a separatory funnel three times using 25 mL methylene chloride. The resulting residues were removed from the SAL, and concentrated in a radiological fume hood to 2 mL.

Additional cleanup of the extract residue was performed following exchange into hexane. The residues were cleaned by washing with concentrated sulfuric acid. The remaining hexane residues were then analyzed for PCBs.

Instrumentation

Analysis was performed according to the test plan BNFL-TP-29953-027, *Analysis for Pesticides and PCBs by Gas Chromatography Dual Electron Capture Detection*. The instrumentation used for the analysis of PCBs consists of a gas chromatograph equipped with two electron capture detectors (ECD). The analytical instrumentation is identified in Table C.1. Both of the detectors were operated at 320° C. Injections were made on-column onto a 10 m fused silica retention gap, which was split

between two analytical columns: a) 0.32 mm X 30 m CLP I (0.50 µm phase, Restek Corp.) and b) 0.32 mm X 30 m CLP II (0.25 µm film thickness, Restek Corp).

Table C.1. PCB/Pesticides/Analysis Instrumentation

System/Instrument	Manufacturer	Model Number	IA&TE ⁽¹⁾ Number
Chromatograph	Hewlett-Packard	5890	WD 11127

(1) Measuring and Test Equipment

If PCBs are detected at a sufficient concentration, confirmation is performed. The instrumentation used for confirmation is a gas chromatograph/ high-resolution mass spectrometer (GC/HRMS).

Analysis Results

PCB results are presented in Table C.2. PCBs were not detected above the quantitation limit (MDL) for these samples.

Table C.2. PCB Results

Tank Material		AP-101 Supernatant			
CAS #	Sample ID	MDL	00-1701 Proc Blk	00-1701 Sample	00-1701 Duplicate
	Units	µg/L	µg/L	µg/L	µg/L
12674-11-2 53469-21-9	Aroclor 1016/1242	0.2	U	U	U
11104-28-2	Aroclor 1221	0.2	U	U	U
11141-16-5	Aroclor 1232	0.2	U	U	U
12672-29-6	Aroclor 1248	0.2	U	U	U
11097-69-1	Aroclor 1254	0.2	U	U	U
11096-82-5 37324-23-5	Aroclor 1260/1262	0.2	U	U	U
11100-14-4	Aroclor 1268	0.2	U	U	U
	Total PCB	1.4	1.4	1.4	1.4
			% Rec	% Rec	% Rec
	TCX (surrogate)		67.8	86.7	90.2
	DCB (surrogate)		100	80.9	113

U flag = Not detected; results less than MDL

As seen in Table C.3, the BNFL target MRQs have been met for supernatants (after adjusting for density).

Table C.3. Target PCB/Pesticides Minimum Reportable Quantities

CAS #	Compound	Solids Target MRQ ⁽¹⁾ µg/Kg	Supernatant Target MRO (Density = 1.31 g/mL) µg/L
-------	----------	--	---

CAS #	Compound	Solids Target MRQ ⁽¹⁾ µg/Kg	Supernatant Target MRO (Density = 1.31 g/mL) µg/L
All	Polychlorinated Biphenyls	3300	4300

⁽¹⁾ MRQ = Minimum Reportable Quantity as provided by BNFL

QC Evaluation

The sample homogenization process used was not consistent with a procedure that would be employed for low concentration PCB analysis. However, the client requested MRQ of microgram quantities is much greater than a concentration of PCBs where adsorption issues would be of concern such as nanogram quantities. So any potential loss of analyte would be of little consequence. However, this means that if PCBs were present in the sample, the actual detection limit may have been affected and is higher than found in Table C.4. Regardless, the actual detection limit would be substantially below the MRQ, particularly since there is more than three orders of magnitude between the current detection limit and MRQ.

Because limited sample was available, the quantity of sample used for spiking was approximately half of that used for unspiked sample analysis. The Laboratory control sample (LCS) consisted of a spiked blank at a volume similar to that used for the samples (100 mL). The LCS recovery is less than the 80-120 % recommended by USEPA SW-846 method 8082. However, the spiking level employed here is considerably lower than the levels used in the SW-846 method by as much as 25 times. The lower spiking level used here is more consistent with up-to-date regulatory expectations. The recovery does meet the general acceptance criteria of 70-130% referred to in method 8000, which would be applied since the spiking level used here fails outside of the range applied in method 8082.

While no specific surrogate acceptance criteria have been developed for tank matrices, the recoveries are all within recommended initial criteria of 20-150%, and meet USEPA CLP SOW OLM01.8 advisory criteria of 60-150% for water.

Table C.4. AP-101 – PCB Spike Recoveries

	<i>Tank Material</i>	AP-101 Supernatant		
	<i>Sample ID</i>	00-1701		LCS
		MS	MSD	
	<i>Units</i>	%Rec (µg/L)	%Rec (µg/L)	%Rec (ug/L)
CAS #	MDL	0.2	0.2	0.2
11097-69-1	Aroclor 1254	106 (4.3)	96.9 (3.9)	71.8 (1.4)
		% Rec	% Rec	% Rec
	TCX (surrogate)	90.2	94.4	76.1
	DCB (surrogate)	118	115	61.8

Data pertaining to the analysis of these samples is located in the project files. Supporting documentation is located in the files for samples 00-2321 (continuing calibration) and 00-1360 (initial calibration). All analysis QC criteria specified in the test plan BNFL-TP-29953-027, *Analysis for Pesticides and PCBs by Gas Chromatography Dual Electron Capture Detection* were met.

Distribution

No. of Copies		No. of Copies	
OFFSITE		ONSITE	
2	DOE/Office of Scientific and Technical Information	1	<u>British Nuclear Fuels, Limited</u> E. Higginbotham BN-FL
		4	<u>CHG</u> M. E. Johnson (4) H4-10
		22	<u>Pacific Northwest National Laboratory</u> P. R. Bredt P7-25 J. A. Campbell P8-08 O. T. Farmer P8-08 S. K. Fiskum P7-22 L. R. Greenwood P7-22 E. W. Hoppe P8-08 F. V. Hoopes P7-22 D. E. Kurath P7-28 G. J. Lumetta P7-22 G. M. Mong P8-08 E. V. Morrey P7-28 R. T. Ratner P7-22 C. Z. Soderquist P7-22 M. J. Steele P7-22 R. G. Swoboda P7-22 M. W. Urie P7-22 J. J. Wagner P7-22 Technical Report Files (5) K1-06

Appendix A: Tank 241-AP-101 Grab Samples



CH2MHILL
Hanford Group, Inc.

CH2M HILL
Hanford Group, Inc.
P.O. Box 1500
Richland, WA 99352

February 15, 2000

CHG-0000767

Mr. J. J. Short, Contracting Officer
Office of Procurement Services
U.S. Department of Energy
Office of River Protection
Post Office Box 450
Richland, Washington 99352-0450

Dear Mr. Short:

CONTRACT NUMBER DE-AC06-99RL14047; SAMPLE MANAGEMENT DOCUMENT
PACKAGE FOR GRAB SAMPLES FROM TANK 241-AP-101

Reference: Letter, J. J. Short II, ORP, to M. P. DeLozier, CHG, "Contract No. DE-AC06-99RL14047 - Direction to Provide Sample From Tank 241-AP-101 to BNFL Inc. (BNFL) and to Archive Sample at 222-S Laboratory," 00-OPD-0017/0000166, dated January 14, 2000.

As requested in the Reference (1), this letter transmits to the U.S. Department of Energy, Office of River Protection, three copies of a "sample management document package" for grab samples from tank 241-AP-101 on February 8, 2000. In accordance with Reference (1), five grab samples were delivered to the 325 Laboratory and five grab samples are archived at the 222-S Laboratory for use by BNFL Inc. Five samples were also archived at the 222-S Laboratory for use by the Waste Feed Delivery program. The attached sample management document package includes the following information regarding tank 241-AP-101 and the samples obtained: recent waste transfer history for tank 241-AP-101, past sampling and analysis of tank 241-AP-101 wastes, sample location selection, sampling information and chain-of-custody forms for the current sampling event.

If you have any questions, please contact Ms. C. DeFigh-Price, at 373-9596.

Very truly yours,

R. F. Wood, Manager
Contracts Administration
CH2M HILL Hanford Group, Inc.

ldf

Attachment

SECTION V: Sampling Information

Waste in tank 241-AP-101 has been identified as potentially a suitable candidate LAW Envelope A feed. On February 8, 2000, ten 120-mL grab samples were obtained from this tank and provided to BNFL Inc. (BNFL) for analysis and testing to evaluate the waste composition and ability to process the waste. An additional five samples were obtained for the Waste Feed Delivery Program.

Table 1 lists the fifteen grab samples and their locations. All samples were obtained on February 8, 2000 from riser 002. At the time of sampling, all samples were clear, yellow liquids with no solids. Five samples were shipped to the Pacific Northwest National Laboratory (PNNL) 325 Laboratory on February 8, 2000, and ten samples were shipped to the Fluor Hanford (FH) 222-S Laboratory on February 9 and 10, 2000. Chain-of-custody forms for the fifteen samples are provided in Section VI.

Table 1. Tank 241-AP-101 Sampling Information

Sample Number	Actual Sample Elevation (inches) ¹	Receiving Laboratory	Organization
1AP-00-1	400	325	BNFL
1AP-00-2	400	222-S	BNFL
1AP-00-3	400	222-S	WFD
1AP-00-4	290	325	BNFL
1AP-00-5	290	222-S	BNFL
1AP-00-6	290	222-S	WFD
1AP-00-7	190	325	BNFL
1AP-00-8	190	222-S	BNFL
1AP-00-9	190	222-S	WFD
1AP-00-10	100	325	BNFL
1AP-00-11	100	222-S	BNFL
1AP-00-12	100	222-S	WFD
1AP-00-13	10	325	BNFL
1AP-00-14	10	222-S	BNFL
1AP-00-15	10	222-S	WFD

Note:

¹Sample elevation is the distance from the tank bottom to the mouth of the sample bottle. All samples were obtained at the requested sample elevations.

BNFL = BNFL Inc.

WFD = Waste Feed Delivery

CHAIN-OF-CUSTODY RECORD FOR CPO

(1) Sample Number IAP-001		(2) Supervisor/Sampler Robert J. Ruznik		(9) Seal Intact Upon Release? <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No
(3) Tank AP-101	(4) Riser 02	(5) Cask/Pig Serial No. A00460-16		(10) Seal Intact Upon Receipt? <input type="checkbox"/> Yes <input type="checkbox"/> No
(6) Shipment Description:				(11) Seal Number AND Cask/Pig SERIAL Number consistent with this record? (Block 5 & 6b) <input type="checkbox"/> Yes <input type="checkbox"/> No
A. Work Package Number ES-00-00045/b B. Cask/Pig Seal Number 12627 C. Date Sample Collected 2-8-00 D. Time Sample Collected 1143 hr.				(12) Laboratory Comments:
(7) Sampling Data - Lithium Bromide <input type="checkbox"/> Y <input type="checkbox"/> N Amount _____ Concentration _____ - X-Ray <input type="checkbox"/> - Partial Sample <input type="checkbox"/> - Retrieved Partial Sample Stroke Length _____				
(8) Field Comments: - TO BE SHIPPED TO 325 BLDG. 2-7-00 - Clear yellow, NO SOLIDS 2-8-00				
(13) Relinquished By (Sign and PRINT) Robert J. Ruznik		(14) Received By (Sign and PRINT) Mr. Dunnington		(15) Date/Time 2/8/00
(17) Relinquished By (Sign and PRINT) Mr. Dunnington		(18) Received By (Sign and PRINT) William Dunnington Fulbright		(19) Date/Time 2/8/00
(21) Relinquished By (Sign and PRINT)		(22) Received By (Sign and PRINT)		(23) Date/Time 2/8/00
(25) Relinquished By (Sign and PRINT)		(26) Received By (Sign and PRINT)		(27) Date/Time
		(28) Receiver Comments		(28) Receiver Comments

CHAIN-OF-CUSTODY-RECORD FOR CPO

(1) Sample Number 1AP-00-11		(2) Supervisor/Sampler Robert J. Praznik		(9) Seal Intact Upon Release? <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No
(3) Tank AP-101	(4) Riser 02	(5) Cask/Pig Serial No. A00460-1		(10) Seal Intact Upon Receipt? <input type="checkbox"/> Yes <input type="checkbox"/> No
(6) Shipment Description:				(11) Seal Number AND Cask/Pig SERIAL Number consistent with this record? (Block 5 & 6b) <input type="checkbox"/> Yes <input type="checkbox"/> No
A. Work Package Number ES-00-00045/0 B. Cask/Pig Seal Number 12630 C. Date Sample Collected 2-8-00 D. Time Sample Collected 1159hr				(12) Laboratory Comments:
(7) Sampling Data - Lithium Bromide Amount NA Concentration 2.2.00 - X-Ray - Partial Sample Retrieved Partial Sample Stroke Length				
(8) Field Comments: - TO BE DELIVERED TO 325 BLDG. 2-7-00 - clear yellow, no solids 2-8-00				
(13) Relinquished By (Sign and PRINT) Robert J. Praznik		(14) Received By (Sign and PRINT) S. Mc.Derming		(15) Date/Time 2/8/00 1630
(17) Relinquished By (Sign and PRINT) S. Mc.Derming		(18) Received By (Sign and PRINT) Robert J. Praznik		(19) Date/Time 2/5/00 1630
(21) Relinquished By (Sign and PRINT)		(22) Received By (Sign and PRINT)		(23) Date/Time
(25) Relinquished By (Sign and PRINT)		(26) Received By (Sign and PRINT)		(27) Date/Time
				(28) Receiver Comments

CHAIN-OF-CUSTODY RECORD FOR CPO

(1) Sample Number IAP-00-7		(2) Supervisor/Sampler Robert J. Frazee		(9) Seal Intact Upon Release? <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	
(3) Tank AP-101	(4) Riser 02	(5) Cask/Pig Serial No. A00460-10		(10) Seal Intact Upon Receipt? <input type="checkbox"/> Yes <input type="checkbox"/> No	
(6) Shipment Description:				(11) Seal Number AND Cask/Pig SERIAL Number consistent with this record? (Block 5 & 6b) <input type="checkbox"/> Yes <input type="checkbox"/> No	
A. Work Package Number ES-00-0004510 B. Cask/Pig Seal Number 126033 C. Date Sample Collected 2-8-00 D. Time Sample Collected 1211hr				(12) Laboratory Comments:	
(7) Sampling Data - Lithium Bromide <input type="checkbox"/> Y <input type="checkbox"/> N Amount NA Concentration 2.7-00 - X-Ray <input type="checkbox"/> - Partial Sample <input type="checkbox"/> Retrieved Partial Sample Stroke Length _____					
(8) Field Comments: - TO BE DELIVERED TO 325 BLDG. 2-7-00 - yellow clear, no solids 2-8-00					
(13) Relinquished By (Sign and PRINT) Robert J. Frazee		(14) Received By (Sign and PRINT) V. Matthews		(15) Date/Time 2-8-00	(16) Receiver Comments
(17) Relinquished By (Sign and PRINT) V. Matthews		(18) Received By (Sign and PRINT) V. Matthews		(19) Date/Time 2-8-00	(20) Receiver Comments
(21) Relinquished By (Sign and PRINT)		(22) Received By (Sign and PRINT)		(23) Date/Time	(24) Receiver Comments
(25) Relinquished By (Sign and PRINT)		(26) Received By (Sign and PRINT)		(27) Date/Time	(28) Receiver Comments

CHAIN-OF-CUSTODY RECORD FOR CPO

(1) Sample Number 1AP-00-10		(2) Supervisor/Sampler Robert J. Jankowski		(9) Seal Intact Upon Release? <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No
(3) Tank AP-101	(4) Riser 02	(5) Cask/Pig Serial No. A004601-7		(10) Seal Intact Upon Receipt? <input type="checkbox"/> Yes <input type="checkbox"/> No
(6) Shipment Description:				(11) Seal Number AND Cask/Pig SERIAL Number consistent with this record? (Block 5 & 6b) <input type="checkbox"/> Yes <input type="checkbox"/> No
A. Work Package Number EA-00-00045/2 B. Cask/Pig Seal Number 1210316 C. Date Sample Collected 2-8-00 D. Time Sample Collected 1222hr				(12) Laboratory Comments:
(7) Sampling Data - Lithium Bromide: <input type="checkbox"/> Y <input type="checkbox"/> N Amount: NA Concentration: 2.7.00 - X-Ray: <input type="checkbox"/> - Partial Sample: <input type="checkbox"/> Retrieved Partial Sample Stroke Length: _____				
(8) Field Comments: - Deliver to 325 Lab @ 3:00 AM - 2-7-00 - yellow clear, no solids 2-8-00				
(13) Relinquished By (Sign and PRINT) Robert Jankowski		(14) Received By (Sign and PRINT) V. Matthews		(15) Date/Time 2-8-00
(17) Relinquished By (Sign and PRINT) V. Matthews		(18) Received By (Sign and PRINT) V. Matthews		(19) Date/Time 2-8-00
(21) Relinquished By (Sign and PRINT)		(22) Received By (Sign and PRINT)		(23) Date/Time
(25) Relinquished By (Sign and PRINT)		(26) Received By (Sign and PRINT)		(27) Date/Time
				(28) Receiver Comments

CHAIN-OF-CUSTODY RECORD FOR CPO

(1) Sample Number IAP-00-13		(2) Supervisor/Sampler Robert J. Puzich		(9) Seal Intact Upon Release? <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No
(3) Tank AE101	(4) Riser 02	(5) Cask/Pig Serial No. 400460-15		(10) Seal Intact Upon Receipt? <input type="checkbox"/> Yes <input type="checkbox"/> No
(6) Shipment Description:				(11) Seal Number AND Cask/Pig SERIAL Number consistent with this record? (Block 5 & 6b) <input type="checkbox"/> Yes <input type="checkbox"/> No
A. Work Package Number ES-00-00045/0 B. Cask/Pig Seal Number 12639 C. Date Sample Collected 2-8-00 D. Time Sample Collected 1235h				(12) Laboratory Comments:
(7) Sampling Data - Lithium Bromide <input checked="" type="checkbox"/> Y <input type="checkbox"/> N Amount NA 2.7.00 Concentration 0.0 - X-Ray <input type="checkbox"/> - Partial Sample <input type="checkbox"/> Retrieved Partial Sample Stroke Length _____				
(8) Field Comments: Deliver to 325 LAKES 300 AREA - GAG 2.7.00 - Chan yellow, no solids. 2-8-00				
(13) Relinquished By (Sign and PRINT) Robert Puzich		(14) Received By (Sign and PRINT) V.P. Matthews		(15) Date/Time 2-8-00
(17) Relinquished By (Sign and PRINT) V. Matthews		(18) Received By (Sign and PRINT) Matthews		(19) Date/Time 2-8-00
(21) Relinquished By (Sign and PRINT)		(22) Received By (Sign and PRINT)		(23) Date/Time
(25) Relinquished By (Sign and PRINT)		(26) Received By (Sign and PRINT)		(27) Date/Time
				(24) Receiver Comments L
				(28) Receiver Comments

Appendix B: Test Instructions

PNNL Test Instruction

Document No.: 29953-81

Rev. No.: 0

Document Control: Only the original
signed copy is controlled

Title: Density Measurements on As-Received AP-101 Samples

Work Location: Radiochemical Processing
Laboratory

Page 1 of 3

Author: Paul Bredt

Effective Date: Upon Final Approval
Supersedes Date: New

Use Category Identification: Reference

Identified Hazards:

- ☐ Radiological
- ☐ Hazardous Materials
- ☐ Physical Hazards
- ☐ Hazardous Environment
- ☐ Other:

Required Reviewers:

- ☒ Technical Reviewer ~~☒ Project Manager~~
- ☒ Building Manager ~~☒ RPL Manager~~
- ☐ Radiological Control ☐ SFO Manager
- ☐ ES&H
- ☒ Quality Engineer

PRB
2/10/00

Are One-Time Modifications Allowed to this Procedure? ☒ Yes ☐ No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? ☐ Yes or ☒ No

FOR REVISIONS:

Is retraining to this procedure required? ☐ Yes ☒ No

Does the OJT package associated with this procedure require revision to reflect procedure changes?
☐ Yes ☐ No ☒ N/A

Approval

Signature

Date

Author

Paul Bredt

2/10/00

Technical Reviewer

D E Kurath

2/10/00

Density Measurements on As-Received AP-101 Samples

Scope

This test plan defines density measurements on samples of tank 241-AP-101 supernatant received from the Hanford tank farms on 2/8/00. These test instructions provides specific details to RPG staff regarding the implementation of Technical Procedure 29953-010, "Measurement of Physical and Rheological Properties of Solutions, Slurries and Sludges".

Justification of Use Category

This test instruction is reference use. Reference use was selected as the use category since this analysis is not a complex process and there are no safety impacts to the order of events. In addition, we may wish to modify the order of analyses or eliminate some analyses depending on the needs at the time of the operation.

Applicability

This test plan applies to RPL staff performing work on BNFL Privatization samples under Project 29953.

Work with actual samples is to be performed in the SAL hot cells by staff under the direction of a cognizant scientist.

Prerequisites

- 1) Keep the sample in a sealed glass container as much as possible to prevent it from drying and reduce the potential for organic contamination.
- 2) Cross-contamination between samples and contamination of samples from outside sources must be minimized at each step. Use new tools and bottles for each sample as much as possible. Those tools which are reused should be washed and rinsed prior to reuse.
- 3) Secondary containment is to be used whenever practical to minimize sample loss in the event of a spilled sample or broken sample bottle.

Quality Control

Quality control has been implemented in Technical Procedure 29953-010, "Measurement of Physical and Rheological Properties of Solutions, Slurries and Sludges". This work is to be conducted under the quality requirements of the Standards-Based Management System (SBMS).

M&TE List

✓ Balance 1: Calib ID 360-0601-016 Calib Exp Date 2/01 Location Cell 2/SAL

Masses of 7 similar Jars to 1AP samples from Julie Johnston of LMHC, Payroll #55327

PR Bredt
02/10/00

Test Instruction 29953-081
Page 3 of 3

1) 125.7 g
2) 124.9 g
3) 125.8 g
4) 124.5 g
 $\bar{x} = 125.22 \text{ g}$

- 1) Weight five new clean volumetric flasks (between 5 and 25 ml) labeled as indicated below.

1AP-00-1	1AP-00-4	1AP-00-7
Tare <u>19.2593</u> g	Tare <u>19.2709</u> g	Tare <u>19.3125</u> g
1AP-00-10	1AP-00-13	
Tare <u>19.3396</u> g	Tare <u>19.4298</u> g	

- 2) Weigh samples 1AP-00-1, 1AP-00-4, 1AP-00-7, 1AP-00-10, and 1AP-00-13. Record the masses.

1AP-00-1	1AP-00-4	1AP-00-7
Mass _____ g	Mass _____ g	Mass _____ g
1AP-00-10	1AP-00-13	
Mass _____ g	Mass _____ g	

High end balance
is broken
PRB
2/11/00

- 3) Fill the volumetrics with supernatant for the respective jars. Record the new mass of the full volumetrics.
used new glass transfer pipet for each sample

1AP-00-1	1AP-00-4	1AP-00-7
Total <u>51.4983</u> g	Total <u>51.5504</u> g	Total <u>51.5914</u> g
Tare <u>19.2593</u> g	Tare <u>19.2709</u> g	Tare <u>19.3125</u> g
Slurry <u>32.2390</u> g	Slurry <u>32.2795</u> g	Slurry <u>32.2789</u> g
Volume <u>25.00</u> ml	Volume <u>25.00</u> ml	Volume <u>25.00</u> ml
$\rho = 1.2896 \text{ g/ml}$	$\rho = 1.2912 \text{ g/ml}$	$\rho = 1.2912 \text{ g/ml}$
1AP-00-10	1AP-00-13	
Total <u>52.3296</u> g	Total <u>52.6236</u> g	
Tare <u>19.3396</u> g	Tare <u>19.4298</u> g	
Slurry <u>32.9900</u> g	Slurry <u>33.1938</u> g	
Volume <u>25.00</u> ml	Volume <u>25.00</u> ml	
$\rho = 1.3196 \text{ g/ml}$	$\rho = 1.3278 \text{ g/ml}$	

- 4) Return the supernatant to the respective jars.
- 5) Weigh samples 1AP-00-1, 1AP-00-4, 1AP-00-7, 1AP-00-10, and 1AP-00-13. Record the masses.

1AP-00-1	1AP-00-4	1AP-00-7
Mass _____ g	Mass _____ g	Mass _____ g
1AP-00-10	1AP-00-13	
Mass _____ g	Mass _____ g	

High end
Balance is
Broken PRB
2/14/00

Work done on 2/11/00
PRB

PNNL Test Instruction

Document No.: BNFL-TP-29953-83

Rev. No.: 0

Document Control: Only the original
signed copy is controlled

Title: AP-101 Homogenization and Subsampling

Work Location: Shielded Analytical
Laboratory/Radiochemical Processing
Laboratory

Page 1 of 5

Author: Paul Bredt

Effective Date: Upon Final Approval

Use Category Identification: Reference

Supersedes Date: New

Identified Hazards:

- ☐ Radiological
- ☐ Hazardous Materials
- ☐ Physical Hazards
- ☐ Hazardous Environment
- ☐ Other:

Required Reviewers:

- | | |
|--|---|
| <input checked="" type="checkbox"/> Technical Reviewer | <input checked="" type="checkbox"/> Project Manager |
| <input checked="" type="checkbox"/> Building Manager | <input checked="" type="checkbox"/> RPL Manager |
| <input type="checkbox"/> Radiological Control | <input type="checkbox"/> SFO Manager |
| <input type="checkbox"/> ES&H | |
| <input checked="" type="checkbox"/> Quality Engineer | |
- not required R B 4/4/00*

Are One-Time Modifications Allowed to this Procedure? ☒ Yes ☐ No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? ☐ Yes or ☒ No

FOR REVISIONS:

Is retraining to this procedure required? ☐ Yes ☒ No

Does the OJT package associated with this procedure require revision to reflect procedure changes?

☐ Yes ☐ No ☒ N/A

Approval

Signature

Date

Author Paul Bredt 3/31/00Technical Reviewer D E Kurath 3/31/00Project Manager D E Kurath 3/31/00SFO Manager Rich J. Hill FOR RE THORNTON 4/3/00

AP-101 Homogenization and Subsampling

Scope

This test plan defines work to be conducted on five AP-101 liquid samples delivered to Battelle from the Hanford tank farms on 2/8/00. Under this Test Plan, the five samples will be combined into a single jar, stirred and then subsampled for other tasks.

Applicability

This test plan applies to RPL staff performing work on BNFL Privatization samples under Project 29953.

Work will be performed in the Shielded Analytical Laboratory (SAL) of the Radiochemical Processing Laboratory (RPL) by staff under the direction of a cognizant scientist.

Prerequisites

- 1) Keep the sample in a sealed glass container as much as possible to prevent it from drying and reduce the potential for organic contamination.
- 2) Cross-contamination between samples and contamination of samples from outside sources must be minimized at each step. Use new tools and bottles for each sample as much as possible. Those tools which are reused should be washed and rinsed prior to reuse.
- 3) Secondary containment is to be used whenever practical to minimize sample loss in the event of a spilled sample or broken sample bottle.
- 4) Since organic analyses will be performed on some of this material, staff are to avoid contacting the samples with plastics. The exception to this is Teflon which is already a sealing material already present in the sample lids.
- 5) This material may contain regulated levels of PCBs. Handle the samples and segregate all wastes accordingly.
- 6) If a balance is not available for this testing, skip the weighing steps.

Quality Control:

This work is to be conducted under the quality requirements of the Standards-Based Management System (SBMS).

PR Bredt
03/31/00

Test Plan: BNFL-TP-29953-083
Page 3 of 5

M&TE List:

_____ Balance 1:

Calib ID 360-06-01-040

Calib Exp Date 2/01

Location 201

If a Category 1 balance is not available, conduct a performance check on an available balance. If needed, user calibrate an available balance and record calibration data below. Data from a user calibrated balance will be limited to information only use.

*In cell Balance is a sartorius max 424g
property # WA 82993*

4/4/00 used 100.003g check weight, reading was 100.004g

Work Instructions:

Note:

This is a very high visibility task within DOE-RL. Secondary containment should be used whenever and wherever possible to prevent inadvertent sample loss and/or hot cell contamination. Great care should be taken during sample transfers.

- 1) Weigh a clean ~1L jar labeled "AP-101 COMP".

AP-101 COMP
Tare 535 g ← Balance 360-06-01-043
EX 8/2000

- 2) Weigh a clean Teflon stir bar.

Stir bar
Tare 7.0498 g ← using Balance 360-06-01-040

- 3) Add the Teflon stir bar to "AP-101 COMP".
- 4) Weigh the sample jars listed below. Transfer all material from the jars to "AP-101 COMP". Reweigh the empty jars and record the masses in the space provided.

Sample Label	Mass (Full)	Mass (Empty)	Mass Transferred
1AP-00-1	300.308	125.973	
1AP-00-4	296.724	126.494	
1AP-00-7	294.544	124.805	
1AP-00-10	299.031	125.430	
1AP-00-13	298.009	125.777	

*all samples were clear yellow with no observable solids
or separable organics layers. PRB 5/23/00*

- 5) Set up secondary containment such that if the primary sample jar (AP-101 COMP) breaks during stirring then the sample material can be easily and cleanly recovered. It is preferred that this be done by placing "AP-101 COMP" in a secondary container that will still allow for proper operation of the stir bar. If this is not possible, then the magnetic stir plate can be placed in secondary containment.

- 6) Securely replace the lid on "AP-101 COMP" and stir the material in "AP-101 COMP" for a minimum of 30 minutes using the magnetic stirrer. Consult with the cognizant scientist on the appropriate stir rate. *started stirring @ 9:31 am 4/4/00 stopped @ 10:25 am*
- 7) Weigh a volume graduated 120 ml glass jar labeled "AP-101 GL".

AP-101 GL

Tare 133.7723 g *vs. 360006-01-040*
133.9021 *PRB 4/4/00*

- 8) Transfer 100 ml (± 10 ml) of supernatant from "AP-101 COMP" to "AP-101 GL". Securely replace the lid on "AP-101 COMP", and "AP-101 GL". Record the mass and volume of material in "AP-101 GL".

AP-101 GL

Total 263.164 g
Tare 133.9021 g
Liquid 129.243 g
Volume 101 ml

*very approximate
↓ due to unknown error
of bottle volume
~ $\rho = 1.2796$*

- 9) Store both "AP-101 COMP", and "AP-101 GL" in secondary containment in a location where they are unlikely to be disturbed.

*all work completed during
morning of 4/4/00
Paul Bredt
4/4/00*

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:

Signature

Print Name

Phone

DEAN E KURATH

4/4/00

DEAN E KURATH

376-6752

MSIN

P7-25

PNNL Project #:

29953

Charge Code:

W 54906

Date Required:

5-12-00

Matrix Type Information

- ♦ Liquids: ☒ Aqueous ☐ Organic ☐ Multi-phase
♦ Solids: ☐ Soil ☐ Sludge ☐ Sediment
☐ Glass ☐ Filter ☐ Metal
☐ Smear ☐ Organic ☐ Other
♦ Other: ☐ Solid/Liquid Mixture, Slurry
☐ Gas ☐ Biological Specimen

If sample matrices vary, specify on Request Page

Disposal Information

Disposition of Virgin Samples:

Virgin samples are returned to requestor unless archiving provisions are made with receiving group!

If archiving, provide:

Archiving Reference Doc # _____

Disposition of Treated Samples:

Dispose ☒ *after 60 days* Return _____

QA/Special Requirements

♦ QA Plan:

SBMS _____

HASQARD (CAWSRP) ☒

♦ Additional QA Requirements? No _____

or Reference Doc # *SEE TABLE 4 ATTACHMENT 1 & 2*

♦ Field COC? *INDIVIDUAL RPLS* Yes ☒

♦ Lab COC Required? No ☒

Yes _____

♦ Hold Time: None ☒

or RCRA _____

CERCLA _____

Other, Specify _____

& Date Sampled _____

Time Sampled _____

♦ Special Storage Requirements:

None ☒ Refrigerate (4°C) _____

or Other, specify _____

♦ Data Quality Review Required? No ☒

Yes _____

Waste Designation Information

♦ Sample Information Check List Attached? Yes ☒

or Reference Doc # _____

or Previous ASR # _____

or Previous RPL ID # _____

Does the Waste Designation Documentation Indicate Presence of PCBs?

No _____

Yes ☒

SEE ATTACHMENT 1 & 2

SICL

Additional or Special Instructions

SEE ATTACHMENT 1 & 2

Send Report To

D. E. KURATH

Phone

376-6752

Phone _____

Preliminary results requested, as available? No _____

Yes ☒

(requesting preliminary results may increase cost)

Receiving and Login Information (to be completed by laboratory staff)

Date Delivered:

RESIDENT IN SAL

Delivered By (optional) _____

Time Delivered (optional) _____

Group ID (optional) _____

CMC Waste Sample? No ☒

Yes _____

Cost Estimate, if requested: \$ _____

Received By: _____

ASR Number:

00-01701

RPL Numbers:

5778

RPG/CMC Work Accepted By:

MW Thue

Signature/Date:

MW Thue 4/3/00

Analytical Service Request (ASR)
(REQUEST PAGE ----- Information Specific to Individual Samples)

(REQUEST PAGE ----- Information Specific to Individual Samples)

[illegible]

(4) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ____; On Attachment ____]

ASR 5778

AP101 OK 6/6/00

00-01701 ~~C-104~~ Supernatant Composite

Digestion-128	SAL	-- Use W54906
ICP-211-CMC	LAB	-- Use CMC WP Number
ICP/MS	ADV INORG	-- Use W54906
GEA-381/474-CMC	RAD	-- Use CMC WP Number
Alpha/Gross-4001/408-CMC	RAD	-- Use CMC WP Number
Beta/Gross-4001/408-CMC	RAD	-- Use CMC WP Number
Am,Cm/AEA-417/422-CMC	RAD	-- Use CMC WP Number
Pu/AEA-417/422-CMC	RAD	-- Use CMC WP Number
U/KPA-4014-CMC	RAD	-- Use CMC WP Number
Sr-90-476/408-CMC	RAD	-- Use CMC WP Number
Se79-440/474-CMC	RAD	-- Use CMC WP Number
Tc99-432/408-CMC	RAD	-- Use CMC WP Number
Direct Sub-Sample		
Density/Solution	SAL	-- Use W54906
IC-212-CMC	LAB	-- Use CMC WP Number
TOC/TIC-381-CMC	LAB	-- Use CMC WP Number
TOC/TIC-380-CMC	LAB	-- Use CMC WP Number
NH3-ISE	LAB	-- Use W54906
CN/Total	LAB	-- Use W54906
H3-418/474-CMC	RAD	-- Use CMC WP Number
C14-381/474-CMC	RAD	-- Use CMC WP Number
Hg-131/201-CMC	LAB	-- Use CMC WP Number
OH-/Titration-228-CMC	LAB	-- Use CMS WP Number
IC-Organic	ORG	-- Use W54906
Ext-Solvent (PCB/Prep)	SAL	-- Hold (Use W54906)
PCB/Pesticide (PCB only)	ORG	-- Hold (Use W54906)

Special Instructions

See Attached Table 2, Table 3 and Table 4 for analyte list, MRQs, and QC Parameters

All analyses are to be done in Triplicate.

Results are to be reported in ug/ml or uCi/ml, as applicable

QC is to include process blank, matrix spike, and LCS (or blank spike)

Method detection limit is to be reported for all analytes.

Digestion:

Prepare one set of triplicate digestions using the sample quantity defined by the procedure (i.e., total of 60 ml). This set of digestions (without further dilutions) will be distributed for all radiochemistry. Additional dilutions may be required due to dose. No MS prepared, where required by method the radionuclides are to be post-spiked.

Prepare a second set of triplicate digestions using 5 ml sample sizes and additional MS (i.e., total 20 ml). This set of digestions will be distributed for ICP (10 ml each) and ICP/MS (15 ml each) analyses. Additional dilutions may be required due to dose. The MS is to include only TCLP metals, all other analytes are to be post-spiked.

Estimated Subsample quantities: (Total ml. including MS) May need to be diluted due to dose

Density/Solution (perform in volumetrics and reuse sample for other analyses)

IC-212-CMC (5 ml)	IC-Organic (5 ml)
TOC/TIC-381-CMC (3 ml)	TOC/TIC-380-CMC (3 ml)
NH3-ISE (4 ml) - No subsample for MS required	CN/Total (8 ml)
H3-418/474-CMC (5 ml)	C14-381/474-CMC (3 ml)
Hg-131/201-CMC (1 ml)	OH-/Titration-228-CMC (5 ml)

PCB Extraction/Analysis: Hold until authorization to proceed has been obtained from D.E. Kurath.

Hot Cell Instructions for Preparation of PCB Organic Samples

- 1) Transfer 100-ml aliquot (40-ml aliquot for MS and MSD) of the AP-101 Comp into a separatory funnel. (Note: if 300 mls are available after all other subsamples have been taken, then use 50-ml aliquots for MS and MS duplicate)
- 2) Add the surrogate spiking solution to all samples (including blank) and the target compound spiking solution to the MS and MSD. Use the entire contents of the vial(s) provided for spiking. After transferring the contents of the spiking vial to the sample, add approximately 0.2 mL of methylene chloride to the vial(s) and transfer this rinsate to the sample. (Note: surrogate and spiking solutions are to be supplied by E. W. Hoppe)
- 3) Perform three sequential separatory funnel shakeout extractions of the supernatant using 25 mL portions of methylene chloride. Collect and combine the three extracts in a 250-mL amber bottle labeled AP-101-PCB-X

Where X = S for sample, D for duplicate, MS for matrix spike and MSD for matrix spike duplicate.

- 4) Dry extracts with anhydrous sodium sulfate. Volume reduction will be performed outside the hot cells.

For further guidance and questions, contact Eric W. Hoppe, (509) 376-2126.

Table 2 Supernate Analyses Required by Contract Specification 7 and Opportunistic Analytes		
Analyte	Minimum Reportable Quantity	Analysis Method
	$\mu\text{g/ml}$	
Al	7.5E+01	ICP-AES
Ba	2.3E+00	
Ca	1.5E+02	
Cd	7.5E+00	
Cr	1.5E+01	
Fe	7.5E+01	
K	7.5E+02	
La	2.3E+00	
Na	1.7E+02	
Ni	3.0E+01	
Pb	3.0E+02	
U	7.8E+02	Kin. Phosphorescence
U	7.8E+02	
Ag	1.7E+01	ICP-AES (Opportunistic)
Bi	1.7E+02	
Cu	1.7E+01	
Mg	1.7E+02	
Mn	1.7E+01	
Nd	1.7E+02	
P	3.3E+02	
Pd	3.9E+02	
Rh	1.8E+01	
Ru	3.6E+01	
Si	9.0E+01	
Sr	1.7E+01	
Ti	1.7E+01	
Zn	1.7E+01	
Zr	1.7E+01	
TIC	1.5E+02	Furnace Oxidation
TOC	1.5E+03	
TOC	1.5E+03	Silver catalyze persulfate and furnace oxidation method
TIC	1.5E+02	Silver catalyze persulfate and furnace oxidation method
Hg	1.5E+00	Cold Vapor AA
Cl	3.0E+02	
F	1.5E+02	IC
NO ₂	2.3E+03	
NO ₃	3.0E+03	
PO ₄	2.3E+03	
SO ₄	2.3E+03	
Formate	To be determined by method	IC (Opportunistic)
Oxalate	1.8E+03	
Acetate	To be determined by method	
Citrate	To be determined by method	

Table 2 Supernate Analyses Required by Contract Specification 7 and Opportunistic Analytes		
Analyte	Minimum Reportable Quantity	Analysis Method
	$\mu\text{Ci/ml}$	
^{237}Np	3.9E-05	ICP-MS
^{237}Pu	3.0E-02	
^{241}Pu	3.0E-02	
^{235}Tc	1.5E-03	
	$\mu\text{g/ml}$	
As	2.3E+00	ICP-MS (Opportunistic)
B	2.3E+00	
Be	2.3E+00	
Ce	2.3E+00	
Co	2.3E+00	
Cs (total)	1.5E+00	
Eu (total)	6.0E+01	
I (natural ^{127}I)	To be determined by method	
Li	2.3E+00	
Mo	2.3E+00	
Pr	2.3E+00	
Rb	2.3E+00	
Sb	2.3E+00	
Se	2.3E+00	
Ta	2.3E+00	
Te	2.3E+00	
Th	2.3E+00	
Tl	2.3E+00	
V	2.3E+00	
W	2.3E+00	
	$\mu\text{Ci/ml}$	
^{241}AMU	To be determined by method	
^{242}AMU	To be determined by method	
^{243}AMU	To be determined by method	
^{231}Pa	7.9E-05	
^{233}U	4.2E-04	
^{234}U	1.2E-04	
^{235}U	4.5E-08	
^{238}U	1.4E-06	
^{239}U	7.2E-08	
^{113}Sn	6.0E-03	
^{129}I (or GEA method)	1.8E-05	
^{90}Sr	3.0E-02	Separations / Liquid Scintillation
^{99}Tc	1.5E-03	Separations / Liquid Scintillation (Opportunistic)
^{236}Pu	1.0E-02	Separations / AEA
^{237}Pu	1.0E-02	
^{238}Pu	5.1E-02	
^{241}Am	3.0E-02	
^{242}Cm	1.5E-01	
^{243}Cm	1.5E-01	

Table 2 Supernate Analyses Required by Contract Specification 7 and Opportunistic Analyses		
Analyte	Minimum Reportable Quantity	Analysis Method
	$\mu\text{Ci/ml}$	
^{154}Eu	$2.0\text{E-}02$	Extended Counting Time GEA
^{152}Eu	$9.0\text{E-}02$	
^{60}Co	$2.1\text{E-}03$	
^{137}Cs	$3.9\text{E-}01$	
	$\mu\text{Ci/ml}$	
$^{106}\text{Ru/Rh}$	To be determined by method	Extended Counting Time GEA (Opportunistic)
^{125}Sb	$1.7\text{E+}00$	
^{134}Cs	$3.9\text{E-}02$	
^{135}Cs	$3.9\text{E-}02$	
^{151}Eu	To be determined by method	
^{241}Am	$1.0\text{E-}02$	
total and free OH	$7.5\text{E+}04 \mu\text{g/ml}$	Titration
Wt% Centrifuged Solids	0.5 wt%	Gravimetry
Wt% Oven Dried Solids	0.25 wt%	Gravimetry
Separate Organic Phase	N/A	Visual Observation
Density	0.9 gm/ml	

Table 3 Additional Supernate Analyses Required by Waste Feed Staging DQO and LAW/HLW Feed Processing DQO (PNNL-12163)		
Analyte	Minimum Reportable Quantity	Analysis Method
	$\mu\text{g/ml}$	
NH_3/NH_4	$1.4\text{E+}02$	ISE
CN	$4.5\text{E+}00$	Distillation / Colorimetric
Total Alpha	$2.3\text{E-}01 \mu\text{Ci/ml}$	Alpha Counting
^3H	$2.1\text{E-}02 \mu\text{g/ml}$	Separations / Liquid Scintillation
^{14}C	$7.2\text{E-}04 \mu\text{Ci/ml}$	Separations / Liquid Scintillation
^{75}Se	$9.0\text{E-}05 \mu\text{Ci/ml}$	Separations / Liquid Scintillation
Total Beta	To be determined ($\mu\text{Ci/ml}$)	Beta Counting
Sum of Alpha	To be determined ($\mu\text{Ci/ml}$)	Summation of Pu-238, Pu-239 + 240, Am-241, Cm-242, Cm-243 + 244

Table 4. Quality Control Parameters for Liquid Analysis

Liquid Fraction ^(a)	Analytical Technique	QC Acceptance Criteria		
		LCS % Recovery ^(b)	Spike % Recovery ^(c)	Duplicate RSD ^(d)
Ag, Al, Bi, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Nd, Ni, P, Pb, Pd, Rh, Ru, S, Sr, Si, Ti, U, Zn, Zr	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	80 - 120%	75 - 125%	<5%
As, B, Ba, Be, Ce, Co, La, Li, Mo, Pr, Rb, Sb, Se, Ta, Te, Th, Tl, V, W, mass unit 90 ^(e)	ICP/MS	80 - 120%	70 - 130%	<15%
Cl ⁻ , F ⁻ , NO ₂ ⁻ , NO ₃ ⁻ , PO ₄ ³⁻ , SO ₄ ²⁻	IC	80 - 120%	75 - 125%	<15%
CN ⁻	Distillation colorimetric	80 - 120%	75 - 125%	<15%
Cs ⁽²⁾ , Eu ⁽³⁾	ICP/MS	N/A	N/A	N/A
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH ₃ /NH ₄ ⁺	ISE, standard additions	80 - 120%	75 - 125%	<15%
OH ⁻	Potentiometric titration	80 - 120%	N/A	<15%
TC/CO ₂ ⁻	Persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
TOC ^(h)	Silver catalyzed persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
Y	Derived from calculation	N/A	N/A	N/A
³ H	Separation/liq. Scintillation	80 - 120%	N/A ⁽ⁱ⁾	<15%
¹⁴ C	Separation/liq. Scintillation	80 - 120%	75 - 125%	<15%
⁶⁰ Co ^(j)	GEA	NP	N/A ^(k)	<15%
⁷⁵ Se ^(l)	Liq. scintillation	NP	N/A ⁽ⁱ⁾	<15%
⁹⁰ Sr	Isotopic specific separation/beta count	75 - 125%	N/A ⁽ⁱ⁾	<15%
⁹⁹ Tc	ICP/MS	80 - 120%	70 - 130%	<15%
⁹⁹ Tc (pertechnetate) ^(l)	Separation/beta count	80 - 120%	70 - 130%	<15%
¹²⁵ Sb	GEA	to be obtained		
¹²⁶ Sn	ICP/MS	80 - 120%	70 - 130%	<15%
¹²⁹ I	ICP/MS or Separation/GEA	NP	N/A ^(k)	<15%
¹³⁷ Cs	GEA	NP	N/A ^(k)	<15%
¹⁵² Eu ⁽ⁱ⁾	GEA	NP	N/A ^(k)	<15%

Table 4. Quality Control Parameters for Liquid Analysis (contd)

Liquid Fraction ⁽¹⁾	Analytical Technique	QC Acceptance Criteria		
		LCS % Recovery ⁽²⁾	Spike % Recovery ⁽³⁾	Duplicate RSD ⁽⁴⁾
¹⁵⁴ Eu ⁽¹⁾	GEA	NP	N/A ⁽⁵⁾	<15%
¹⁵⁵ Eu ⁽¹⁾	GEA	NP	N/A ⁽⁵⁾	<15%
²³¹ Pa ⁽¹⁾	ICP/MS	Developed by Laboratory		
²³³ U	ICP/MS	90 - 110%	75 - 125%	<15%
²³⁴ U	ICP/MS	90 - 110%	75 - 125%	<15%
²³⁵ U	ICP/MS	90 - 110%	75 - 125%	<15%
²³⁶ U	ICP/MS	90 - 110%	75 - 125%	<15%
²³⁸ U	ICP/MS	80 - 120%	70 - 130%	<15%
²³⁷ Np ⁽¹⁾	ICP/MS	90 - 110%	75 - 125%	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
²³³ Pu, ²³⁹ Pu, ²⁴⁰ Pu ^{(1), (m)}	Separation/AEA	NP	N/A ⁽¹⁾	<15%
²⁴¹ Pu/Am, ²⁴² Pu ^(m)	ICP/MS	80 - 120%	70 - 130%	<15%
²⁴¹ Am ^(m)	Separation/AEA	NP	N/A ⁽¹⁾	<15%
²⁴² Cm ^(m)	Separation/AEA	NP	N/A ⁽¹⁾	<15%
²⁴³ Am/Cm ^(m)	ICP/MS	90 - 110%	75 - 125%	<15%
²⁴³ - ²⁴⁴ Cm ^(m)	Separation/AEA	NP	N/A ⁽¹⁾	<15%
Total Alpha ⁽¹⁾	Proportional counter	70 - 130%	70 - 130%	<15%
Total Beta	Beta counting	70 - 130%	70 - 130%	<15%
Total Gamma	GEA-Sum of isotopes	N/A	N/A	N/A
Density		N/A	N/A	N/A
Wt% dissolved solids ⁽⁶⁾	Gravimetric	80 - 120%	N/A	<21%

Acronyms:

- AEA - Alpha Energy Analysis
- CVAA - Cold Vapor Atomic Absorption
- GEA - Gamma Energy Analysis
- IC - Ion Chromatography
- ICP/AES - Inductively Coupled Plasma Atomic Emission Spectroscopy
- ICP/MS - Inductively Coupled Plasma Mass Spectroscopy
- LSC - Laboratory Control Standard
- N/A - Not applicable
- NP - Not performed
- RSD - Relative Standard Deviation
- Wt% - Weight percent

Table 4. Quality Control Parameters for Liquid Analysis (cont'd)

Liquids footnotes

- (a) Analytes for the Solubility Screening Test are a subset of this analyte list. Refer to Section 7.6.3.
- (b) LCS = Laboratory Control Standard. This standard is carried through the entire method. The accuracy of a method is usually expressed as the percent recovery of the LCS. The LCS is a matrix with known concentration of analytes processed with each preparation and analyses batch. It is expressed as percent recovery; i.e., the amount measured, divided by the known concentration, times 100.
- (c) For some methods, the sample accuracy is expressed as the percent recovery of a matrix spike sample. It is expressed as percent recovery; i.e., the amount measured less the amount in the sample, divided by the spike added, times 100. One matrix spike is performed per analytical batch. Samples are batched with similar matrices.
For other analytes, the accuracy is determined based on use of serial dilutions as described in Section 7.7.2.2.
- (d) RSD = Relative Standard Deviation between the samples. Sample precision is estimated by analyzing replicates taken separately through preparation and analysis. Acceptable sample precision is usually <15% RSD if the sample result is at least 10 times the instrument detection limit.
$$RSD = (\text{standard deviation of the mean/mean}) \times 100$$
- (e) ICP-MS mass unit 90 includes ^{90}Sr , ^{90}Y , and ^{92}Zr .
- (f) Analysis required for only liquid fraction.
- (g) Total Cs and Eu are sums of all isotopes, therefore spiking and LCS does not apply.
- (h) Analysis for oxalate may be required as described in Section 7.5.
- (i) Matrix spike analyses are not required for this method because a tracer is used to correct for analyte loss during sample preparation and analysis. The result generated using the tracer accounts for any inaccuracy of the method on the matrix. The reported results reflect this correction.
- (j) An extended counting time in the presence of high ^{137}Cs activity may be required to achieve the minimum reportable quantity for ^{60}Co and ^{154}Eu , ^{153}Eu .
- (k) The measurement is a direct reading of the energy and the analysis is not affected by the sample matrix; therefore, a matrix spike is not required.
- (l) The sum of ^{238}Pu , ^{239}Pu , ^{240}Pu , and ^{241}Am activities will be used as a measurement of alpha-emitting TRU when total alpha measurement in the liquid fraction is equal to or exceeds 6.0E-05 Ci/L for Envelopes A and B and 4.0E-04 Ci/L for Envelope C. The selected trigger values correspond to 70% of the LAW envelope limits for TRU. The selected isotopes account for greater than 95% of the alpha-emitting TRU activity based on previous analysis of Phase I candidate tank waste (Esch 1997a, 1997b, 1997c). Additional isotopes which are defined as alpha-emitting TRU (e.g., ^{237}Np , ^{242}Pu , ^{243}Cm , ^{243}Am , and $^{243-244}\text{Cm}$) are not used to calculate total TRU activity because the MDAs for these isotopes are large in comparison with the envelope limits and it is expected that their concentrations are well below the MDA. Note that ^{241}Pu is a beta-emitting TRU whose analysis, along with ^{242}Cm , is required specifically for class C waste determination.
- (m) Analysis for these analytes is required only if the total alpha measurement is equal to or exceeds 6.0E-05 Ci/L for Envelope A and B feed and 4.0E-04 Ci/L for Envelope C. These values correspond to 70% of the envelope limit for TRU. Based on the previous analysis results for Phase I tanks, at total alpha values less than specified, the values for these analytes are less than the MDA. Therefore, if the total alpha values are less than specified, the MDA for the individual analyte will be used to fulfill the data needs for this DQO.
- (n) Weight percent dissolved solids method is described in Section 7.3.4.

RPL Sample Information Checklist

(Please have client or sample owner complete to the best of their knowledge or provide the information obtained from the client.)

CLIENT
Name: D. E. KURATH
Phone: 376-6752
Employer: BATTELLE

CHECKLIST PREPARED BY:
Print Name: M. W. YRIZ
Signature: [Signature]
Date: 4/3/00

(To be complete by Battelle research or laboratory staff)

Client Sample Number(s)	Description (Use page 2 if more than 4 samples)	RPL Unique Identifier(s)
<u>AP-101 COMP</u>	<u>AP-101 TANK WASTE COMPOSITE</u>	<u>00-01701</u>

1. Is the sample from a commercial chemical product, manufacturing chemical intermediate, or off-specification commercial chemical product (i.e., P or U Listed)? (See 40 CFR 261.33) ☐ Yes ☒ No
If yes, what chemicals or waste codes? _____

2. Is the sample from an F or K listed source? (See 40 CFR 261.31 and 261.32). ☒ Yes ☐ No
If yes, what is the source and waste code? HANFORD TANK WASTE

3. Mark any of the following physical and chemical characteristics or constituents (and associated waste code) you suspect to be present:

- | | | | |
|--|--|--|--|
| <input type="checkbox"/> Explosive (D003) | <input type="checkbox"/> Pyrophoric (D003) | <input type="checkbox"/> Shock Sensitive (D003) | <input type="checkbox"/> Generates Toxic Gases in Water (D003) |
| <input type="checkbox"/> Oxidizer (D001) | <input type="checkbox"/> Asbestos | <input type="checkbox"/> Peroxide Former (D003) | <input type="checkbox"/> Halogenated Hydrocarbons (WP01 or WP02) |
| <input type="checkbox"/> Sulfides (D003) | <input type="checkbox"/> Cyanides (D003) | <input type="checkbox"/> Corrosive Solids (WSC2) | <input type="checkbox"/> Polycyclic Aromatic Hydrocarbons (WP03) |
| <input type="checkbox"/> Water Reactive (D003) | <input type="checkbox"/> Air Reactive (D003) | <input type="checkbox"/> Corrosive to Steel (D002) | |
| <input checked="" type="checkbox"/> Corrosive (D002); pH = <u>12.5 or less</u> | <input type="checkbox"/> Ignitable (D001); Flashpoint = _____ °F or _____ °C | | |

4. Mark any of the following constituents (and associated waste code) you suspect to be present (40 CFR 261.24):

- | | | | |
|---|--|--|---|
| <input type="checkbox"/> Arsenic (D004) | <input type="checkbox"/> Benzene (D018) | <input type="checkbox"/> 2,4-Dinitrotoluene (D030) | <input type="checkbox"/> Nitrobenzene (D036) |
| <input type="checkbox"/> Barium (D005) | <input type="checkbox"/> Carbon tetrachloride (D019) | <input type="checkbox"/> Endrin (D012) | <input type="checkbox"/> Pentachlorophenol (D037) |
| <input type="checkbox"/> Cadmium (D006) | <input type="checkbox"/> Chlordane (D020) | <input type="checkbox"/> Heptachlor (& its epoxide) (D031) | <input type="checkbox"/> Pyridine (D038) |
| <input checked="" type="checkbox"/> Chromium (D007) | <input type="checkbox"/> Chlorobenzene (D021) | <input type="checkbox"/> Hexachlorobenzene (D032) | <input type="checkbox"/> Tetrachloroethylene (D039) |
| <input checked="" type="checkbox"/> Lead (D008) | <input type="checkbox"/> Chloroform (D022) | <input type="checkbox"/> Hexachlorobutadiene (D033) | <input type="checkbox"/> Toxaphene (D015) |
| <input type="checkbox"/> Mercury (D009) | <input type="checkbox"/> Cresol, o-, m-, or p- (D023-D025) | <input type="checkbox"/> Hexachloroethane (D034) | <input type="checkbox"/> Trichloroethylene (D040) |
| <input type="checkbox"/> Selenium (D010) | <input type="checkbox"/> 2,4-D (D016) | <input type="checkbox"/> Lindane (D013) | <input type="checkbox"/> 2,4,5-Trichlorophenol (D041) |
| <input type="checkbox"/> Silver (D011) | <input type="checkbox"/> 1,4-Dichlorobenzene (D027) | <input type="checkbox"/> Methoxychlor (D014) | <input type="checkbox"/> 2,4,6-Trichlorophenol (D042) |
| | <input type="checkbox"/> 1,2-Dichloroethane (D028) | <input type="checkbox"/> Methyl ethyl ketone (D035) | <input type="checkbox"/> 2,4,5-TP (Silvex) (D017) |
| | <input type="checkbox"/> 1,1-Dichloroethylene (D029) | | <input type="checkbox"/> Vinyl chloride (D043) |

5. List any known RCRA Underlying Hazardous Constituents: None known

6. Are there any state hazardous waste codes associated with the sample? ☐ Yes ☒ No
If yes, please list: _____

7. Is the sample known to contain >2 ppm PCBs? ☐ Yes ☐ No
If yes, is the sample concentration: ☐ <50 ppm ☐ >50 ppm?
If <50 ppm, what is the source of PCBs (if known)? PCB CONCENTRATION UNKNOWN. HOWEVER, CONSIDER SAMPLE AS HAVING PCBs FROM SOURCE >50ppm. SEE ATTACHED LETTER

8. Is the sample radioactive? ☒ Yes ☐ No
If yes, list any known isotopes, activities, or dose rates associated with the sample:

See Attachment for best estimate of Radionuclide Concentrations

(To be completed by Battelle research or laboratory staff)

Battelle Project #: 24953

Charge Code #: W54906

Signature: _____ Date: _____

Receiving Inspection	
No Anomalies Found	<input type="checkbox"/>
Anomalies Found, RPL POC Notified	<input type="checkbox"/>

Best TWINS estimate of contents of AP-101 for use with SICL

Analyte	Units	AP-101
Al	g/L	9.66E+00
Bi	g/L	0.00E+00
Ca	g/L	3.87E-02
Cl	g/L	1.32E+00
TIC as CO3	g/L	1.40E+01
Cr	g/L	1.08E-01
F	g/L	2.29E+00
Fe	g/L	3.87E-03
Hg	g/L	0.00E+00
K	g/L	1.79E+01
La	g/L	0.00E+00
Mn	g/L	0.00E+00
Na	g/L	1.46E+02
Ni	g/L	6.30E-03
NO2	g/L	4.03E+01
NO3	g/L	1.31E+02
OHTOTAL	g/L	6.95E+01
Pb	g/L	3.13E-03
PO4	g/L	9.94E-01
Si	g/L	8.39E-02
SO4	g/L	3.43E+00
Sr	g/L	3.29E-06
TOC	g/L	3.15E+00
UTOTAL	g/L	2.53E-02
Zr	g/L	1.74E-02

Analyte	Units	AP-101
3H	mCi./L	1.38E-02
14C	mCi./L	1.77E-04
59Ni	mCi./L	3.29E-04
60Co	mCi./L	3.29E-03
63Ni	mCi./L	3.29E-02
79Se	mCi./L	3.87E-04
90Sr	mCi./L	1.35E-01
90Y	mCi./L	1.35E-01
93Zr	mCi./L	2.90E-03
93mNb	mCi./L	2.10E-03
99Tc	mCi./L	5.12E-02
106Ru	mCi./L	7.92E-06
113mCd	mCi./L	1.52E-02
125Sb	mCi./L	2.55E-02
126Sn	mCi./L	1.25E-02
129I	mCi./L	5.56E-05
134Cs	mCi./L	2.55E-03
137Cs	mCi./L	1.73E+02
137mBa	mCi./L	1.64E+02
151Sm	mCi./L	2.08E+00
152Eu	mCi./L	7.74E-04
154Eu	mCi./L	7.32E-02
155Eu	mCi./L	4.05E-02
226Ra	mCi./L	7.60E-08
227Ac	mCi./L	1.48E-07
228Ra	mCi./L	4.42E-05
229Th	mCi./L	1.04E-06
231Pa	mCi./L	6.65E-07
232Th	mCi./L	6.00E-06
232U	mCi./L	3.64E-06
233U	mCi./L	1.40E-05
234U	mCi./L	1.15E-05
235U	mCi./L	4.42E-07
236U	mCi./L	8.27E-07
237Np	mCi./L	1.82E-04
238Pu	mCi./L	1.07E-05
238U	mCi./L	8.39E-06
239Pu	mCi./L	1.44E-04
240Pu	mCi./L	3.43E-05
241Am	mCi./L	1.95E-04
241Pu	mCi./L	9.96E-04
242Cm	mCi./L	3.20E-07
242Pu	mCi./L	4.15E-09
243Am	mCi./L	1.80E-08
243Cm	mCi./L	1.62E-05
244Cm	mCi./L	3.54E-04



River Protection Project
Waste Treatment Plant
3000 George Washington Way
Richland, WA 99352
Tel: (509) 371-3500
Fax: (509) 371-3504

Kathleen Whelan
MSIN K9-84
Battelle Memorial Institute
Pacific Northwest National Laboratory
902 Battelle Boulevard
P.O. Box 999
Richland, Washington 99352

Direct tel: 509-375-4312
Direct fax: 509-372-4334
CCN#: 012259

March 17, 2000

Dear Kathy:

Contract No. DE-AC06-96RL13308 - W375-SC-98-4168 - REQUEST FOR PROPOSAL
TO CONDUCT PCB ANALYSIS OF TANK 241-AP-101 SAMPLE

References: 1. CCN 012160, Letter M. E. Johnson, BNFL Inc., to Eugene Morrey,
Battelle, "Request for Proposal to Conduct Revised Analysis of Tank
241-AP-101 Samples", dated March 15, 2000.

BNFL Inc. received verbal notification on March 15, 2000 from Dr. N. R. Brown, U.S.
Department of Energy Office of River Protection (DOE-ORP), the tank 241-AP-101 sample
currently at Battelle facilities may have in part originated from a waste source that contained
greater than 50ppm polychlorinated biphenyl (PCB) compounds. Battelle personnel should
conduct appropriate handling practices for this waste sample, consistent with Battelle
procedures.

BNFL Inc. is requesting Battelle to prepare a proposal for conducting analysis of the tank 241-
AP-101 sample to determine the concentration of PCB compounds following EPA *Test
Methods for Evaluation of Solid Waste Physical / Chemical Methods*, SW-846. The requested
PCB analysis of the tank 241-AP-101 sample is to be conducted in addition to the analyses
already requested by BNFL Inc. in correspondence number 012160 (ref.).

RECEIVED
CONTRACT SERVICES

MAR 20 2000

Please indicate in your proposal the volume of additional tank 241-AP-101 sample (if necessary) for conducting the PCB analyses. Please submit your proposal to Ms. Anne Thompson by March 24, 2000. Please contact Michael Johnson, if you have technical inquiries relating to this request for proposal

Sincerely,


James O. Knight
Systems Acquisition Manager

ANT/vle

cc:

D. Blumenkranz
J. R. Cook
M. E. Johnson
Contracts File
Contracts Correspondence File
PDC

Neil R. Brown
Karyn Wiemers

BNFL Inc.
BNFL Inc.
BNFL Inc.
BNFL Inc.
BNFL Inc.
BNFL Inc.

ETC-1/P-122
ETC-1/P113
B-267

DOE-O
DOE-O

H6-60
H6-60

Test Instruction

Unique Numerical Designation: TI-29953-086

Revision number: 0

Effective Date: April 17, 2000

Controlling Procedure No: N/A

Author Approval: *Singh P. J. 4/17/00*

Technical Reviewer: *J. E. Kuntz 4/17/00*

BNFL Reviewer: N/A

TITLE: AP-101 Precipitation/Crystallization Versus Temperature

1. Set bath temperature to 10°C
2. Inspect the AP-101 sample (sample ID: AP-101 GL) for precipitates

6/6/00

Notes: Appears to be some solids floating around in the AP-101 GL sample.

Will filter, then transfer filtrate to clean glass bottle

→ "AP-101 GL Filter"

3. If the sample has solids, filter through a 0.45-µm nylon membrane. If there are no solids, proceed directly to step 4.
4. Place the sample in the cooling bath at 10°C *PLACED 1040 6/8/00 MRZ*
5. Inspect the sample for solids each day (excluding weekends) for a period of seven days; note observations below

Date	Time	Observations
6-9-00	1040	NO VISIBILITY CHANGES
6-12-00	1230	NO VISIBLE CHANGES
6-13-00	1530	NO visibility changes
6-14-00	-	NOT TAKEN
6-15-00	(AP) NO	visible changes

TEMP.	INT.
9°C	MRZ tare wt = 133.5504
9°C	MRZ Balance Cal. info
10°C	MRZ 384-06-01-004
-	MRZ Due 8/2000
9°C	MRZ (END OF TEST)

Note

If no solids have formed after seven days, proceed to step 17.

6. Label a 120-mL glass jar as AP101-10°-Liquid

7. Weigh vial AP101-10°-Liquid

Wt. AP101-10°-Liquid = _____ g (7a)

Note balance calibration information:

Calibration ID: _____

Calibration Date: _____

Due Date: _____

Note

During steps 8 through 11, keep AP-101 GL in the 10°C water bath to the extent possible to avoid warming the sample

8. Using a pipette transfer the liquid from AP-101 GL to AP101-10°-Liquid; take care not to transfer any solids during this process

9. Weigh vial AP101-10°-Liquid

Wt. AP101-10°-Liquid = _____ g (9a)

Wt. liquid = 9a – 7a = _____ g (9b)

10. Place a piece of filter paper into AP-101 GL and tip the jar so that the excess liquid wicks up the filter paper; take care not to remove any solids during this process

11. Remove the filter paper

12. Weigh vial AP-101 GL with the damp solids

Wt. AP-101 GL = _____ g (12a)

13. Calculate the weight of the damp solids

Wt. wet solids = 12a – 133.9021 = _____ g (13a)

Note: The tare weight of AP-101 GL is 133.9021 g

14. Dry the solids in AP-101 GL to a constant weight at 105°C

Notes:

15. Weigh vial AP-101 GL with the dry solids

Wt. AP-101 GL = _____ g (15a)

16. Calculate the weight of the dry solids

Wt. solids = 15a - 133.9021 = _____ g (16a)

17. End of Test

Appendix C: Analytical Reports

Shielded Analytical Laboratory Bench Sheet

Client: MW URIE

WP Number: W54906

TI#/ASR: ASR 5778

Procedure: Bench Instruction

AP101 DENSITIES

SAMPLE IDENTIFICATION

LAB NUMBER	SAMPLE IDENT.	FLASK TARE WT (g)	FLASK GROSS WEIGHT (g)	FLASK SIZE (mL)	DENSITY (g/mL)
00-01701	AP-101 COMP	9.3824	22.4570	10	1.307
00-01701-DUP	AP-101 COMP	9.4763	22.5629		1.309
00-01701-TRI	AP-101 COMP	9.5085	22.5902		1.308

$$\text{DENSITY} = \frac{\text{FLASK GROSS (g)} - \text{FLASK TARE (g)}}{\text{FLASK SIZE (mL)}}$$

M&TE: ☒ Cell 2 (360-06-01-016) Mettler AE160 Balance Other _____

_____ Cell 5 (360-06-01-019) Sartorius LP4200S Balance

_____ Bench (510-06-01-014) Mettler AT201 Balance

_____ Bench (360-06-01-040) Denver A160 Balance

Analyst:

Date:

Reviewer:

Date:

[Signature]

4-5-00

[Signature]

4/7/00

**Battelle PNNL/RPG/Inorganic Analysis ...
ICPAES Data Report**

Project: 29953 / W54906
Client: D. Kurath

ACL Number(s): 00-01701

Client ID: "AP-101-COMP"

ASR Number: 5778

Total Sample: 1

Procedure: PNL-ALO-211, "Determination of Elements by Inductively Coupled
Argon Plasma Atomic Emission Spectrometry" (ICP-AES).

Analyst: D.R. Sanders

Analysis Date (Filename): 04-11-00 (A0595) [ALO-128 SAL/vh]

See Chemical Measurement Center 98620: ICP-325-405-1 File for Calibration and
Maintenance Records.

M&TE Number: ICPAES instrument -- WB73520
Mettler AT400 Balance -- Ser.No. 360-06-01-029

Derry Wagner 5-17-00
Reviewed by

M. Kurath 5-17-00
Concur

5/17/00

Page 1

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

ASR-5778

Sample **AP-101-COMP** (RPL# 00-01701) was prepared in triplicate by the Shielded Analytic Lab (SAL) using ALO-128 acid digestion of liquids procedure. A process blank, blank-spike and matrix-spike were also prepared similarly at the same time as the samples. Approximately 6.6 grams of liquid (density 1.308 g/ml) or about 5ml, was processed and diluted to a final volume of about 25 ml (final solution volume was weighed and density corrected to a volume). Some insoluble precipitate remained after processing requiring filtration before analysis could be performed. Analytical dilution prior to ICPAES analysis required 5, 10 and 50-fold dilution in order to quantify all analytes of interest. Measurement results are reported in $\mu\text{g/ml}$ corrected for sample density, processing and analytical dilution. Volumes and weights have been recorded on bench sheets and included with this report.

Quality control objectives were met for the majority of analytes of interest listed in ASR-5778 attachment 2: page 1 of 6 (Table 2 ...MRQ's) and page 4 of 6 (Table 4 ...QC Acceptance Criteria).

Objectives missed:

- a) Sulfur is not available on the ICPAES instrument used for this work.
- b) MRQ's required for "...Contract Specification 7..." for barium ($2.3 \mu\text{g/ml}$) and lanthanum ($2.3 \mu\text{g/ml}$) were below EQL (2.5 and $12.7 \mu\text{g/ml}$ respectively).
- c) Also, MRQ's for "...Opportunistic Analytes" for rhodium (18), ruthenium (36), and silicon (90) were below EQL (76 , 278 , and $126 \mu\text{g/ml}$ respectively).
- d) LCS for silver (69%R) was below "QC Acceptance Criteria" (80%-120%R).
- e) Spike Recovery for silver (35%R), barium (30%R), and lead (65%R) was below "QC Acceptance Criteria" (75% - 125%R).
- f) Duplicate %RPD (average) for phosphorous (15.8%RPD) was below "QC Acceptance Criteria" (<15%RPD).

See attachment 1 and 2 to this report.

Quality control check-standard results met tolerance requirements for all analytes except as noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements. Please note that MRQ is equivalent to EQL in this report.

Five fold serial dilution:

(liquid samples)

Results were within tolerance limit of $\leq 10\%$ for all analytes tested after correcting for dilution.

5/17/00

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Duplicate RPD (Relative Percent Difference):

(liquid samples) All analytes of interest were recovered within tolerance limit of $\leq 15\%$ relative percent difference (RPD) except as follows. RPD for phosphorous in the three sample replicates ranged from about 15% to 28% RPD. Average RPD for phosphorous within the three replicates was 15.8%. Chromium in the three sample replicates ranged from about 1% RPD to about 16% RPD. Average RPD for chromium within the three replicates was about 8.9%.

Post-Spiked Samples (Group A):

(liquid samples) All analytes of interest were recovered within tolerance of 75% to 125% except silicon. Silicon recovery (147%) exceeded tolerance limit. All other analytes of interest above EQL were within tolerance.

Post-Spiked Samples (Group B):

(liquid samples) All analytes of interest were recovered within tolerance of 75% to 125% except palladium. Palladium recovery (53%) exceeded tolerance limit. All other analytes of interest above EQL were within tolerance. Palladium was found low in the group B spike. Single element palladium at 2 $\mu\text{g/ml}$ measured at the beginning, middle and end of the run ($2.00 \pm 0.04 \mu\text{g/ml}$) were well within tolerance limit. No palladium was detected in the sample.

Blank Spike:

(liquid samples) All analytes of interest were recovered within tolerance limit of 80% to 120% recoveries except silver. Low recovery of silver in the blank spike (about 69%) was probably due to the small amount of hydrochloric acid used during sample preparation resulting in some silver precipitating from solution.

Matrix Spiked Sample:

(liquid samples) All analytes of interest were recovered within tolerance limit of 75% to 125% recoveries except silver, barium and lead. Silver recovery (about 35%), barium recovery (about 30%) and lead recovery (about 65%) were all low and may be related to the presence of sulfate and/or carbonate present in the sample. Low silver recovery is probably, in part, due to the small amount of hydrochloric acid used during sample processing. All sample aliquots had a small amount of precipitate remaining after processing requiring filtration.

Battelle PNNL/RPG/Inorganic Analysis ...

ICPAES Data Report

Quality Control Check Standards (solid and liquid samples):

Concentrations of all analytes were within tolerance limit of $\pm 10\%$ accuracy in standards (except palladium): QC_MCVA, QC_MCVB, and QC_SSTMCV. Calibration Blank (ICP98.0) concentration is acceptable, less than two times IDL. Palladium in QC check standard MCVB was low (about 42% to 48%) however, a single element standard of palladium at 2.0 $\mu\text{g}/\text{ml}$ measured at the beginning, middle and end of the ICP run was well within tolerance limit indicating acceptable palladium accuracy. No palladium was detected in the sample aliquots.

High Calibration Standard Check (solid and liquid samples):

Verification of the high-end calibration for all analytes measured is within tolerance limits of $\pm 5\%$ accuracy.

Process Blank:

(liquid samples)

All analytes of interest were within tolerance limit of $\leq \text{EQL}$ or $< 5\%$ of sample concentration in the prepared samples.

Laboratory Control Standard (LCS):

(liquid samples)

The blank-spike and matrix-spike were prepared and measured as an LCS control (See results described above)

Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%.

Comments:

- 1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.
- 2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.
- 3) Routine precision and bias is typically $\pm 15\%$ or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 $\mu\text{g}/\text{mL}$ (0.5 per cent by weight).
- 4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.
- 5) The maximum number of significant figures for all ICP measurements is 2.

Multiplier=		4.8	25.3	25.3	25.0
RPL/LAB #=		00-1701-PB	00-1701 @5	00-1701-DUP @5	00-1701-TRI @5
		AP-101	AP-101	AP-101	AP-101
Client ID=		Comp	Comp	Comp	Comp
		(Supernatan	(Supernatan	(Supernatan	(Supernatan
		t)	t)	t)	t)
Det. Limit	Run Date=	4/11/00	4/11/00	4/11/00	4/11/00
(ug/mL)	(Analyte)	ug/mL	ug/mL	ug/mL	ug/mL
0.025	Ag	--	--	--	--
0.060	Al	4.52	7,380	6,680	6,760
0.250	As	--	--	--	--
0.050	B	6.62	15.0	14.4	15.1
0.010	Ba	--	[0.32]	[0.34]	[0.33]
0.010	Be	--	[1.1]	[1.0]	[1.0]
0.100	Bi	--	--	--	--
0.250	Ca	[1.4]	[7.8]	[7.5]	[7.7]
0.015	Cd	--	[2.0]	[1.9]	[2.0]
0.200	Ce	--	--	--	--
0.050	Co	--	--	--	--
0.020	Cr	--	158	137	135
0.025	Cu	[0.18]	[1.7]	[1.5]	[1.5]
0.050	Dy	--	--	--	--
0.100	Eu	--	--	--	--
0.025	Fe	[0.38]	[2.9]	[2.4]	[2.2]
2.000	K	--	31,700	30,500	31,500
0.050	La	--	--	--	--
0.030	Li	--	--	--	--
0.100	Mg	--	--	--	--
0.050	Mn	--	--	--	--
0.050	Mo	--	[12]	[10.0]	[9.8]
0.150	Na	14.3	132,000	125,000	131,000
0.100	Nd	--	--	--	--
0.030	Ni	1.63	8.62	7.68	7.50
0.100	P	--	371	290	281
0.100	Pb	--	[15]	[15]	[15]
0.750	Pd	--	--	--	--
0.300	Rh	--	--	--	--
1.100	Ru	--	--	--	--
0.500	Sb	--	--	--	--
0.250	Se	--	--	--	--
0.500	Si	[24]	143	132	137
1.500	Sn	--	[60]	--	--
0.015	Sr	--	--	--	--
1.500	Te	--	--	--	--
1.000	Th	--	--	--	--
0.025	Ti	--	--	--	--
0.500	Tl	--	--	--	--
2.000	U	--	[68]	[62]	[56]
0.050	V	--	--	--	--
2.000	W	--	--	--	--
0.050	Y	--	--	--	--
0.050	Zn	[0.91]	[5.8]	[5.5]	[5.4]
0.050	Zr	--	[1.4]	[1.4]	[1.3]

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

ASR-5778 "ICPAES Measurement Results -- Data"

A0595	S.Vol(ml)=	5.0539	5.0060	5.0650	5.0637
4-11-00/drs	F.Vol(mL)=	24.5000	25.3000	25.6000	25.3000
	(PF)	1.0000	1.0000	1.0000	1.0000
	(DF)	1.0000	1.0000	1.0000	1.0000
	(ADF)	1.0000	5.0000	5.0000	5.0000
	Multiplier=	4.8	25.3	25.3	25.0
	RPL/LAB #=	00-1701-PB	00-1701 @5	00-1701-DUP @5	00-1701-TRI @5
		AP-101	AP-101	AP-101	AP-101
		Comp	Comp	Comp	Comp
		(Supernatan	(Supernatan	(Supernatan	(Supernatan
	Client ID=	t)	t)	t)	t)
Det. Limit	Run Date=	4/11/00	4/11/00	4/11/00	4/11/00
(ug/mL)	(Analyte)	ug/mL	ug/mL	ug/mL	ug/mL
0.025	Ag	—	—	—	—
0.060	Al	4.52	7,380	6,680	6,760
0.250	As	—	—	—	—
0.050	B	6.62	15.0	14.4	15.1
0.010	Ba	—	[0.32]	[0.34]	[0.33]
0.010	Be	—	[1.1]	[1.0]	[1.0]
0.100	Bi	—	—	—	—
0.250	Ca	[1.4]	[7.8]	[7.5]	[7.7]
0.015	Cd	—	[2.0]	[1.9]	[2.0]
0.200	Ce	—	—	—	—
0.050	Co	—	—	—	—
0.020	Cr	—	158	137	135
0.025	Cu	[0.18]	[1.7]	[1.5]	[1.5]
0.050	Dy	—	—	—	—
0.100	Eu	—	—	—	—
0.025	Fe	[0.38]	[2.9]	[2.4]	[2.2]
2.000	K	—	31,700	30,500	31,500
0.050	La	—	—	—	—
0.030	Li	—	—	—	—
0.100	Mg	—	—	—	—
0.050	Mn	—	—	—	—
0.050	Mo	—	[12]	[10.0]	[9.8]
0.150	Na	14.3	132,000	125,000	131,000
0.100	Nd	—	—	—	—
0.030	Ni	1.63	8.62	7.68	7.50
0.100	P	—	371	290	281
0.100	Pb	—	[15]	[15]	[15]
0.750	Pd	—	—	—	—
0.300	Rh	—	—	—	—
1.100	Ru	—	—	—	—
0.500	Sb	—	—	—	—
0.250	Se	—	—	—	—
0.500	Si	[24]	143	132	137
1.500	Sn	—	[60]	—	—
0.015	Sr	—	—	—	—
1.500	Te	—	—	—	—
1.000	Th	—	—	—	—
0.025	Ti	—	—	—	—
0.500	Tl	—	—	—	—
2.000	U	—	[68]	[62]	[56]
0.050	V	—	—	—	—
2.000	W	—	—	—	—
0.050	Y	—	—	—	—
0.050	Zn	[0.91]	[5.8]	[5.5]	[5.4]
0.050	Zr	—	[1.4]	[1.4]	[1.3]

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Attachment No. 1 of 2, ASR-5778 "ICPAES Measurement Results -- Statistical Summary"

Multiplier=		4.8	25.3	25.3	25.0			
RPL/LAB #=		00-1701-PB AP-101 Comp (Supernatan t)	00-1701 @5 AP-101 Comp (Supernatan t)	00-1701-DUP @5 AP-101 Comp (Supernatan t)	00-1701-TRI @5 AP-101 Comp (Supernatan t)			
Client ID=		4/11/00	4/11/00	4/11/00	4/11/00			
Det. Limit (ug/mL)	Run Date= (Analyte)	ug/mL	ug/mL	ug/mL	ug/mL	Ave (n=3)	StDev.	%RSD
0.025	Ag	—	—	—	—			
0.060	Al	4.52	7,380	6,680	6,760	6940	383	5.5
0.250	As	—	—	—	—			
0.050	B	6.62	15.0	14.4	15.1	14.83	0.38	2.6
0.010	Ba	—	[0.32]	[0.34]	[0.33]	[0.33]	0.010	3.0
0.010	Be	—	[1.1]	[1.0]	[1.0]	[1.03]	0.06	5.6
0.100	Bi	—	—	—	—			
0.250	Ca	[1.4]	[7.8]	[7.5]	[7.7]	[7.67]	0	2.0
0.015	Cd	—	[2.0]	[1.9]	[2.0]	[1.97]	0	2.9
0.200	Ce	—	—	—	—			
0.050	Co	—	—	—	—			
0.020	Cr	—	158	137	135	143.33	13	8.9
0.025	Cu	[0.18]	[1.7]	[1.5]	[1.5]	[1.57]	0.12	7.4
0.050	Dy	—	—	—	—			
0.100	Eu	—	—	—	—			
0.025	Fe	[0.38]	[2.9]	[2.4]	[2.2]	[2.50]	0.36	14.4
2.000	K	—	31,700	30,500	31,500	31233	643	2.1
0.050	La	—	—	—	—			
0.030	Li	—	—	—	—			
0.100	Mg	—	—	—	—			
0.050	Mn	—	—	—	—			
0.050	Mo	—	[12]	[10.0]	[9.8]	[10.60]	1.2	11.5
0.150	Na	14.3	132,000	125,000	131,000	129333	3786	2.9
0.100	Nd	—	—	—	—			
0.030	Ni	1.63	8.62	7.68	7.50	7.93	0.6	7.6
0.100	P	—	371	290	281	314.00	50	15.8
0.100	Pb	—	[15]	[15]	[15]	[15.00]	—	0.0
0.750	Pd	—	—	—	—			
0.300	Rh	—	—	—	—			
1.100	Pd	—	—	—	—			
0.500	Sb	—	—	—	—			
0.250	Se	—	—	—	—			
0.500	Si	[24]	143	132	137	137.33	5.5	4.0
1.500	Sn	—	[60]	—	—	[60]		
0.015	Sr	—	—	—	—			
1.500	Te	—	—	—	—			
1.000	Th	—	—	—	—			
0.025	Ti	—	—	—	—			
0.500	Tl	—	—	—	—			
2.000	U	—	[68]	[62]	[56]	[62]	6.0	9.7
0.050	V	—	—	—	—			
2.000	W	—	—	—	—			
0.050	Y	—	—	—	—			
0.050	Zn	[0.91]	[5.8]	[5.5]	[5.4]	[5.6]	0.21	3.7
0.050	Zr	—	[1.4]	[1.4]	[1.3]	[1.4]	0.06	4.2

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Attachment No. 2 of 2, ASR-5778 "ICPAES Measurement Results -- Quality Control Parameters"

ASR-5778 D. Kurath A0595 4/11/00 RPL#: 00-1701 Client ID: "AP-101 COMP"

PF= 25.3

Analyte	MRQ (ug/ml)	IDL(ICPAES) (ug/ml)	EQL (ug/ml)	[80%-120%] LCS (%R)	[75%-125%] Spike Rec'y (%R)	[<15%] Dup %RSD (%RSD)
1 Ag	17	0.025	6.3	69	35	--
2 Al	75	0.06	15.2			5.5
3 Ba	2.3	0.01	2.5	87	30	[3]
4 Bi	170	0.1	25.3			--
5 Ca	150	0.25	63.3			[2]
6 Cd	7.5	0.015	3.8	92	87	[2.9]
7 Cr	15	0.02	5.1	93	94	8.9
8 Cu	17	0.025	6.3			[7.4]
9 Fe	75	0.025	6.3			[14]
10 K	750	2	506.0			2.1
11 La	2.3	0.05	12.7			--
12 Mg	170	0.1	25.3			--
13 Mn	17	0.05	12.7			--
14 Na	170	0.15	38.0			2.9
15 Nd	170	0.1	25.3			--
16 Ni	30	0.03	7.6	88	85	7.6
17 P	330	0.1	25.3			15.8
18 Pb	300	0.1	25.3	101	65	[0]
19 Pd	390	0.75	189.8			--
20 Rh	18	0.3	75.9			--
21 Ru	36	1.1	278.3			--
22 Sb	90	0.05	126.5			4
23 Sr	17	0.015	3.8			--
24 Ti	17	0.025	6.3			--
25 U	780	2	506.0			[9.7]
26 Zn	17	0.05	12.7	89	92	[3.7]
27 Zr	17	0.05	12.7			[4.2]

Note: Gray areas indicate quality results outside of control limits of Table 2, "Supernate Analyses Required by Contract Specification 7 and Opportunistic Analytes" and Table 4, "Quality Control Parameters for Liquid Analysis" (reference: See ASR-5778 Attachment 2 page 1 of 6, and page 4 of 6)

Battelle PNNL/RPG/Inorganic Analysis --- IC Report

REVISION 1 (June 13, 2000)

Client:	D Kurath	Charge Code/Project:	W54906/29953
RPL Numbers:	00-1701 (triplicate)	ASR Number:	5778
Analyst:	MJ Steele	Analysis Date:	April 23-24, 2000

Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
M&TE: IC system (WD25214); Balance (360-06-01-031) --- See Chemical Measurement Center 98620 RIDS IC File for Calibration, Standards Preparations, and Maintenance Records.

Final Results:

The AP-101 composite sample (00-1701) was analyzed in triplicate by ion chromatography (IC) for inorganic anions as specified in the governing ASR. The final results are presented in Table 1 below. All analytical samples were diluted 2000 to 5000 fold at the IC workstation to ensure that all anions reported were measured within the calibration range. Per the ASR the samples were analyzed in triplicate and the relative standard deviation between the measurements is reported. The minimum reportable quantity (MRQ) for each anion was defined by the ASR, and all MRQ were met at the dilutions analyzed. From recoveries demonstrated on some of the verifications standards analyzed, the AP-101 sample matrix has an adverse effect on the IC column and some reported results may be 10% to 20% low. By further diluting the sample, this effect could be minimized, but at the expense of meeting the MRQs (particularly phosphate, sulfate, and oxalate).

For IC column and parameters used, the IC system can not separate fluoride, acetate, and formate; the IC system quantifies and reports all as fluoride. It is unlikely that the levels of fluoride quantified are present in the tank waste, and since acetate and formate could be present in the AP-101 sample, the fluoride results should be used with reservation.

Table 1		F (*)	Cl	NO ₂	Br	NO ₃	PO ₄	SO ₄	C ₂ O ₄
RPL Number	Sample ID	ug/ml	ug/ml	ug/ml	ug/ml	ug/ml	ug/ml	ug/ml	ug/ml
	Target MRQs	150	300	2,300	n/a	3,000	2,300	2,300	1,800
00-1701	AP-101 Composite	2,880	2,000	42,200	< 500	133,000	1,040	4,070	1,100
00-1701 Dup	AP-101 Composite Duplicate	2,880	1,920	41,000	< 500	130,000	1,000	3,940	1,060
00-1701 Trip	AP-101 Composite Triplicate	2,940	2,010	42,400	< 500	133,000	1,020	4,080	1,100
	Rel Std Dev (%)	1.2%	2.5%	1.8%	n/a	1.3%	2.0%	1.9%	2.1%
00-1701 MS	AP-101 Composite MS %Rec	114%	111%	119%	108%	128%	119%	108%	121%
	Blank Spike %Rec	110%	107%	105%	108%	78%	104%	106%	107%

Rel Std Dev (5) = Relative Standard Deviation - Sample, Duplicate, Triplicate

MS %Rec = Matrix Spike Standard % Recovery, BS %Rec = Blank Spike Standard % Recovery

(*) Use fluoride results with reservation; IC system can not resolve fluoride, acetate, and formate.

Battelle PNNL/RPG/Inorganic Analysis --- IC Report

Q.C. Comments

Duplicates/Replicates: The ASR requested that the analyses be performed in triplicate and therefore the relative standard deviation was calculated for the three analyses. The Quality Control parameters defined in Table 4 attached to the ASR states acceptance criteria of <15% relative percent difference (RSD). Table 1 presents the RSD based on comparison of the triplicate analysis. The RSD meets the acceptance criteria for all anion.

Matrix Spike: The matrix spike recoveries for all anions, except nitrate, were within the spiking acceptance criteria of 75% to 125% as established by ASR attachment Table 4. The high nitrate concentration of the sample relative to the nitrate spiking level (i.e., sample concentration was 10 times greater than the spike concentration) significantly impacts the ability to measure the nitrate spike. No attempt was made to spike the samples at additional spiking level.

Blank Spike: The blank spike recoveries for all anions, except nitrate, were within the Laboratory Control Samples (LCS) acceptance criteria of 80% to 120% as established by the ASR attachment Table 4. Nitrate recovered slightly low at 78%. It should be noted that the blanks spike nitrate recovery is not consistent with other control standards analyzed during the analysis run (e.g., Verification Check Standards).

System Blank/Processing Blanks: A dozen system blanks were process during the analysis of the slurry samples. No anions were detected in the system blanks above the method detection limit.

Quality Control Calibration Verification Check Standards: Over twenty mid-range verification standards were analyzed throughout the analysis runs. Numerous verification standards analyzed just after the analysis of some of the AP-101 samples demonstrated low recoveries (i.e., recoveries ranging from 80% to 90%). Failure of the verification standard was only observed in those standards analyzed just after the AP-101 samples that had were prepared with the least dilution. Numerous reruns were performed alternating the AP-101 (at various dilutions) and the verification standard to obtain valid data.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested.
- Routine precision and bias are typically $\pm 15\%$ or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Approval: MW this

Date 6-13-00

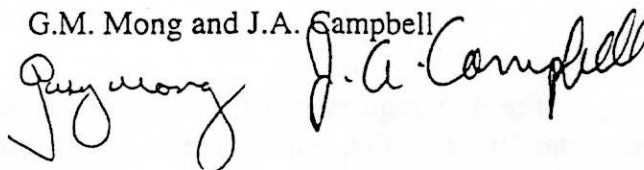
Archive Information:

Files: ASR 5778 Kurath Rev 1.doc

ASR 5729 5764-68 5778.xls

IC Results for oxalate, formate, acetate, and citrate

G.M. Mong and J.A. Campbell



Instrumentation

The analytical instrumentation utilized for the analysis of low-molecular weight organic acids consisted of an ion chromatography unit equipped with a conductivity detector. A Dionex AS-11 column and AG-11 guard column were used at ambient temperature with a 25- μ L sample loop. An anion suppressor was used. The flow rate of the mobile phase was 2.0 mL/min. A description of the IC system is provided in Table 1.a.

Table 1.a Ion Chromatography Instrumentation

System/Instrument	Manufacturer	Model Number	M&TE ⁽¹⁾ Number
Ion Chromatograph	Dionex	500 DX	WD 24293
Conductivity Detector	Dionex	CD20	WD 24295

⁽¹⁾ Measuring and Test Equipment

The IC gradient conditions were: (a) 0.0 min 0% 100 mM NaOH, 98.1% deionized water and 1.9% 5 mM NaOH, (b) 6.4 min 0% 100 mM NaOH, 0% deionized water and 100% 5 mM NaOH, and (c) 18.4 min 35% 100 mM NaOH, 0 % deionized water and 65% 5 mM NaOH. The mobile phase contained a gradient of deionized water and a weak solution of NaOH.

Analysis Results

Samples were analyzed using PNNL operating procedure AOAM-1 (Method for the Analysis and Quantification of Organic Acids in Simulated and Actual Hanford Tank Wastes by Ion Chromatography). Sample AP-101 was done in triplicate at a dilution found to give acceptable total ion loading to the column (Dionex AS-11 anion exchange media). This dilution was calculated to be a 2242 times the sample weight as delivered to the 325 shielded laboratory.

Although acetate is reported, acetate coelutes with glycolate and requires the use of an alternate column for separation from glycolate. Without additional separation, one can not unequivocally state that the acetate/glycolate peak contains only acetate, only glycolate, a combination of both analytes, or a possible contaminant. From tank waste and solubility studies, the results are tank waste type dependent. In other words, for one tank waste the component may be entirely acetate. For another tank with a different fill history, the analytical results may indicate the component may be primarily glycolate or a combination of both analytes (Sharma et al. 1998, Camaioni et al. 1998, Barney 199, Ashby et al. 1994).

The results are shown below for quantity (in micrograms) of acetate, formate, and oxalate found per milliliter of sample., based on a measured density of 1.308 g/mL. Citrate was below detectability in each sample using this dilution scheme.

Sample	μg acet/mL	μg form/mL	μg oxal/mL	μg citrate/mL
# 1	1543	1131	1799	ND
# 2	1793	1256	1781	ND
# 3	1601	1196	1817	ND
Average	1645	1194	1800	ND

The detection limits for acetate, formate, oxalate, and citrate are estimated to be 550 $\mu\text{g/mL}$, 450 $\mu\text{g/mL}$, 890 $\mu\text{g/mL}$, and 890 $\mu\text{g/mL}$, respectively, based on the dilution factors.

QC Evaluation

Matrix spiking was done at a level approximately 1.5 times the level of analyte in the final diluted samples. This level of spiking is at a challenging level for accurate quantitation; the reported levels are near the lowest calibration point for each analyte. These results are summarized below:

MS spike		ms	msd-1	msd-2	average
recovery	Acetate	175%	105%	91%	124%
	Formate	71%	104%	79%	84%
	Oxalate	122%	92%	129%	114%

The value for acetate in the matrix spike sample should be considered suspect, as the peak quantitation had to be done by estimation (since the peak size was so small). The other data represent quantitation which was extracted directly from electronic integration data. Overall, the average data for MS and MSD is within acceptable limits. The LCS consisted of a mixture of formate and oxalate. The per cent recoveries obtained were 89.3 and 103.8%, respectively.

References

1. Sharma, A.K., S.A. Clauss, G.M. Mong, K.L. Wahl, J.A. Campbell. 1998. Analysis and quantification of organic acids in simulated Hanford tank waste and Hanford tank waste. J. of Chromatogr, 805, 101-107.
2. Camaioni, D.M., W.D. Samuels, J.C. Linehan, S.A. Clauss, A.K. Sharma, K.L. Wahl, J.A. Campbell. 1996. Organic Tanks Safety Program, FY 96 Waste Aging Studies, PNNL-11312, Pacific Northwest National Laboratory, Richland, Wa.

3. Ashby, E.C. et al. 1994. Synthetic Waste Chemical Mechanism Studies, WHC-EP-0823, Westinghouse Hanford Company, Richland, Wa.
4. Barney, GL. 1996. Solubilities of Significant Organic Compounds in HLW Tank Supernate Solutions-FY 1996 Progress Report, WHC-EP-0899, Westinghouse Hanford Company, Richland, Wa.

Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report

Client: D. Kurath
 ACL Numbers: 00-1701
 Analyst: MJ Steele

Charge Code/Project: W54906 / 29953
 ASR Number: 5778
 Analysis Date: May 12 and April 13-14, 2000

Procedure: PNL-ALO-381, "Direct Determination of TC, TOC, and TIC in Radioactive Sludges and Liquids by Hot Persulfate Method"
 PNL-ALO-380, "Determination of Carbon in Solids Using the Coulometric Carbon Dioxide Coulometer"

M&TE: Carbon Analysis System (WA92040); Balance (360-06-01-023).

Final Results:

RPL Number	Sample ID	HOT PERSULFATE METHOD						FURNACE METHOD	
		TIC ($\mu\text{gC/ml}$)	TIC RPD	TOC ($\mu\text{gC/ml}$)	TOC RPD	TC ($\mu\text{gC/ml}$)	TC RPD	TC ($\mu\text{gC/ml}$)	TC RPD
00-01701	AP-101 COMPOSITE	6,550		1,860		8,410		8,530	
00-01701	AP-101 COMPOSITE	6,400	2%	1,980	6%	8,390	0%	8,670	2%
00-01701	AP-101 COMPOSITE	6,430		2,030		8,460		8,620	
	RSD	1.2%		4.5%		0.4%		0.8%	
00-01701 MS	MS Recovery	98.5%		96.3%		97.5%		97.5%	
	MDL	40		80		80		170	

RPD = Relative Percent Difference

RSD = Relative Standard Deviation

The analysis of the liquid sample submitted under ASR 5778 was performed by the hot persulfate wet oxidation method and by the furnace oxidation method. The hot persulfate method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95°C for TOC, all on the same sample, with TC being the sum of the TIC and TOC. The furnace oxidation method determines TC by oxidizing all forms of carbon (i.e., inorganic and organic) in oxygen at 1000 °C. Although the ASR requested TOC and TIC by the furnace method, the method is considered reliable only for TC. For the sample matrix analyzed the furnace method and hot persulfate method should provide equivalent TC results, and this was confirmed by the results, with the average hot persulfate TC being 8420 $\mu\text{g/ml}$ and the furnace TC being 8610 $\mu\text{g/ml}$; an RPD of about 2%.

The table above shows the results, rounded to two to three significant figures. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank.

Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report

Q.C. Comments:

The TIC standard is calcium carbonate and TOC/TC standard is α -Glucose (the certificates of purity are attached). The standard materials were used in solid form for system calibration check standards as well as matrix spikes. The QC for the methods involves calibration blanks, system calibration standards, sample duplicates, and one matrix spike per matrix type.

Calibration Standards: The QC system calibration check standards were all within acceptance criteria of 90% to 110%, with the average recovery being about 99% for TIC and 99% for TOC over the two days that the hot persulfate analyses were performed and about 100% for TC for the furnace analyses.

Calibration Blanks: The calibration blanks run at the beginning, middle, and end of the analysis run were acceptable and the standard deviations for the TIC and TOC blanks are near the historical pooled standard deviation used to establish the method detection limits. On the May 12th run, the TOC blanks were about 2 to 3 times higher than normal; however, the blanks were quite consistent and the high blanks should have no effect on the reported data.

Duplicates: No actual sample duplicates were identified on the ASR. However, the relative percent difference (RPD) between replicates (i.e., a sample and duplicate analysis) was less than 10% for TIC, TOC, and TC. Besides the RPD, the relative standard deviation (RSD) for the triplicate analyses was calculated, and all RSDs are less than 5%, indicate very good precision between for all measurements. The RSD met the ASR Table 4 RSD acceptance criterion of <15%.

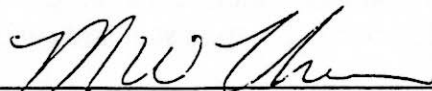
Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The matrix spike for both the hot persulfate and furnace method demonstrated recoveries of about 96% to 99% for TIC, TOC, and TC; well within the acceptance criteria of 75% to 125% recovery.

Laboratory Control Sample: No LCS is included in the carbon analysis procedure.

General Comments:

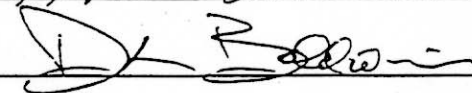
- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- Routine precision and bias are typically $\pm 15\%$ or better for non-complex samples that are free of interferences.
- The estimated quantitation limit (EQL) is defined as 5 times the MDL. Results less than 5 times the MDL have higher uncertainties, and RPDs are not calculated for any results less than 5 times the MDL.
- Some results may be reported as less than (" $<$ ") values. These less than values represent the sample MDL (method detection limit), which is the system MDL adjusted for the volume of sample used for the analysis. The system MDL is based on the attached pooled historical blank data. The evaluation and calculation of the system MDL is included in the data package.

Report Prepared by:



Date 5-18-00

Review/Approval by:



Date 5-22-00

Archive Information:

Files: ASR 5778 Kurath.doc

ASR 5668R 5778 Kurath.xls



Battelle

... Putting Technology To Work

Project 29953

No. _____

Internal Distribution

File/LB

Date May 17, 2000

To K. E. Kurath

From S. K. Fiskum *SK Fiskum*

Subject Hydroxide Analyses for AP101 Composite ASR
5778, Rev. 1

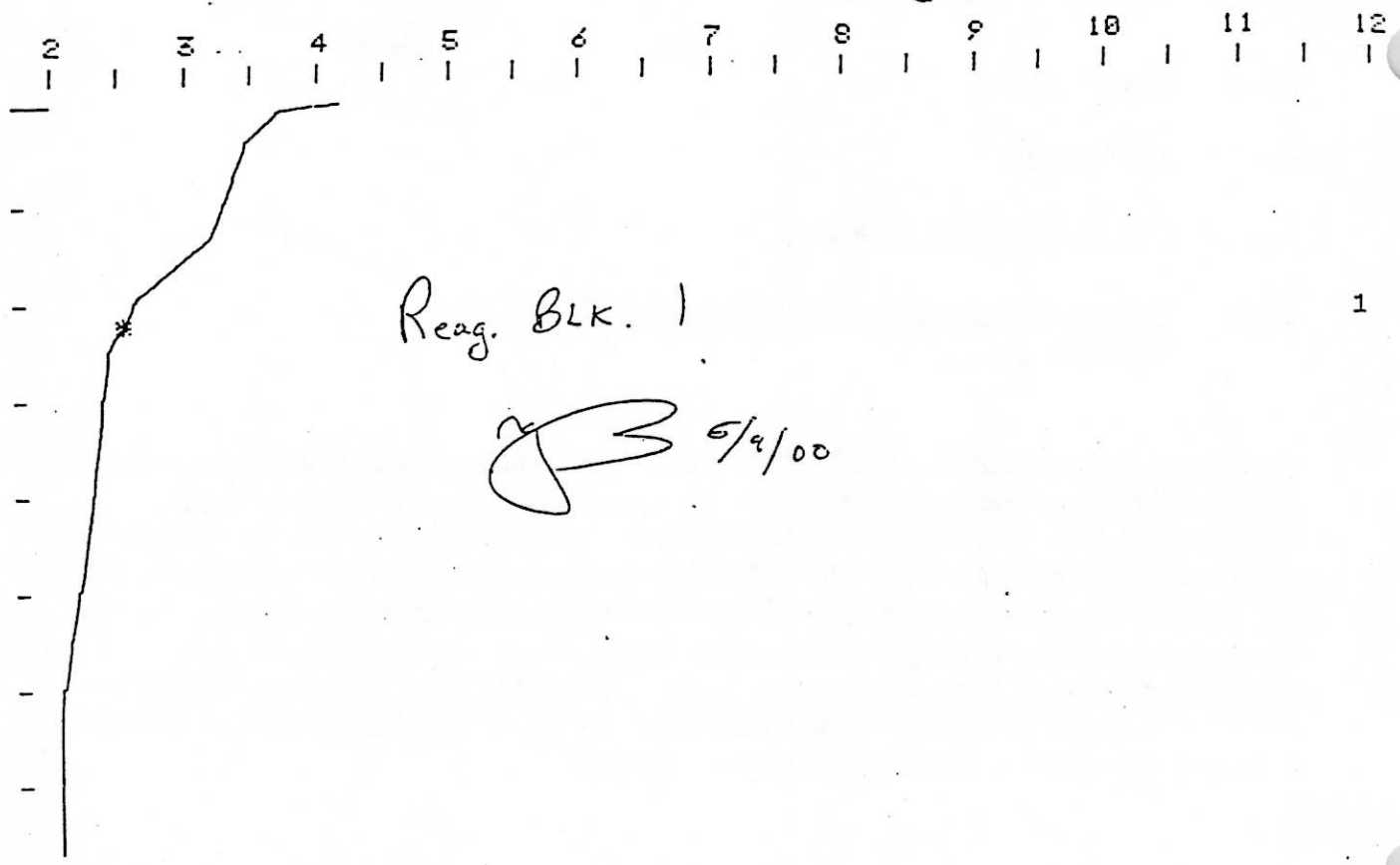
A sample of the composite from tank AP101 (00-1701) was analyzed in triplicate for the hydroxide content following procedure PNL-ALO-228. Direct sample aliquots were analyzed using a Brinkman 636 Auto-Titrator. A 0.1186 N NaOH solution was prepared for use as a standard and sample spike and the titrant was a 0.2040 M HCl prepared solution. Triplicate results (2.49, 2.42, and 2.38 M OH) gave an average OH molarity of 2.43 with a relative standard deviation of $\pm 3\%$. The standard recoveries averaged 100% and the sample spike recovered at 88%. Hydroxide was not detected in the reagent blank, demonstrating an MDC of 0.02M OH or $3.4 \times 10^2 \mu\text{g OH/mL}$. A second titration inflection point was determined at an average pH of 7.5 and a third inflection point at an average pH of 4.9. The titration curves are attached.

Review: LR Greenwood 5/17/00

PH(M) 10.012 4.012 0.012 - 0.024
 Δ PH 0.012 SLOPE 0.881 U(AS)/MV 29.3
 TEMP/C 25.0

1/7/00
[Signature]

0.25ML/DIV V(START)/ML 0.000 PH



Reag. BLK. 1

[Signature] 5/9/00

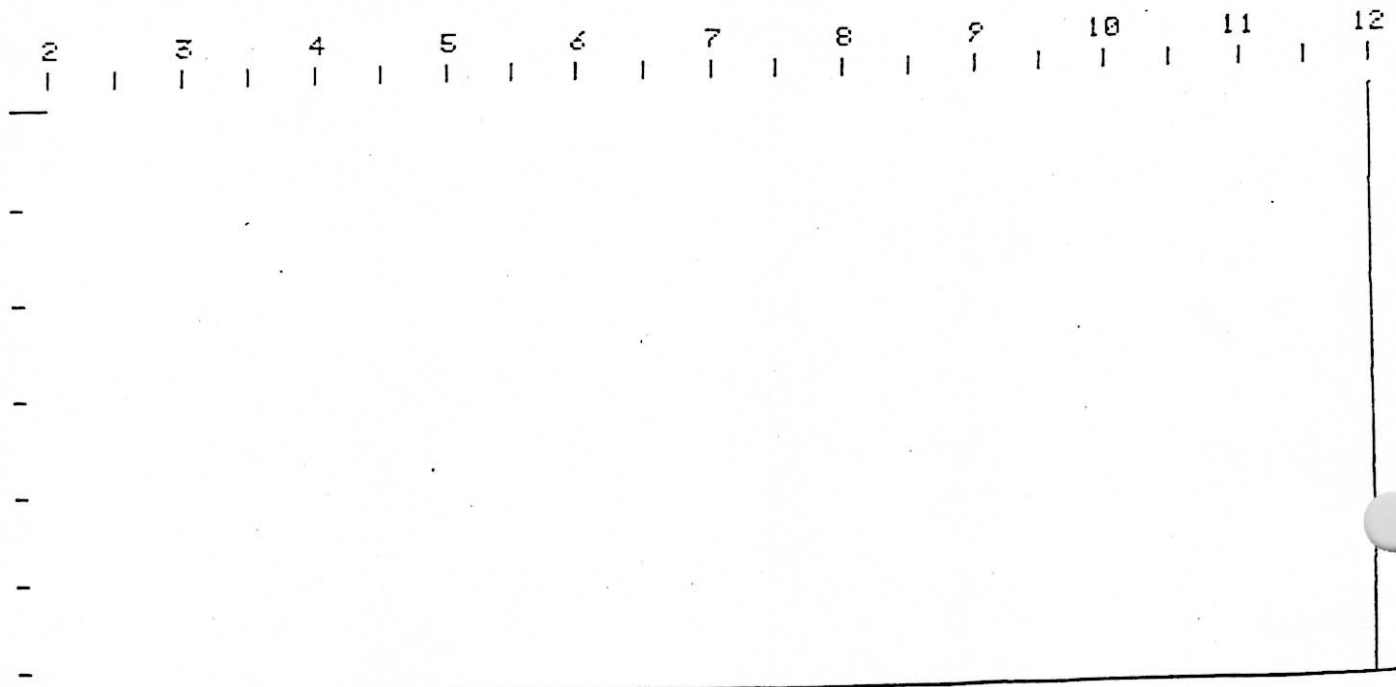
1

BRINKMANN CAT # 2025015-1

ROUTINE # 101
 # 1 PH(INIT) 4.120 V(TE)/ML 2.149
 1 V/ML 0.566 PH(M) 2.513

DATE NAME

0.25ML/DIV V(START)/ML 0.000 PH



OH⁻ Std # 1
5 mL of 0.1186 M NaOH

5/9/00

12

BRINKMANN CAT # 2025015-1

ROUTINE # 101
2 PH(INIT) 12.216 V(TE)/ML 5.000
1 V/ML 2.925 PH(M) 7.992
2 V/ML 2.954 PH(M) 4.511

DATE

NAME

0.25ML/DIV V(START)/ML 0.000 PH

2 3 4 5 6 7 8 9 10 11 12
| | | | | | | | | | | |

OH⁻ Std #2

5 mL of 0.1186 M NaOH

93 5/1/00

ROUTINE # 101
3 PH(INIT) 12.113 V(TE)/ML 5.000
1 V/ML 2.936 PH(M) 7.841
2 V/ML 2.993 PH(M) 4.132

DATE

NAME

0.25ML/DIV V(START)/ML 0.000 PH

2 3 4 5 6 7 8 9 10 11 12
| | | | | | | | | | | |

0.25ML/DIV V(START)/ML 0.000 PH

2 3 4 5 6 7 8 9 10 11 12

ASR 5778

KURATH Sample # 00-1701-1

100 μ L

mg 5/9/00

BRINKMANN CAT # 2025015-1

ROUTINE # 101
7 PH(INIT) 12.074 V(TE)/ML 4.676
1 V/ML 1.219 PH(M) 10.523
2 V/ML 1.728 PH(M) 7.920
3 V/ML 2.081 PH(M) 4.951

0.25ML/DIV V(START)/ML 0.000 PH

2 3 4 5 6 7 8 9 10 11 12

Karath
Duplicate100mL of 00-1701 (2)

5/9/00

193

ROUTINE # 101
8 PH(INIT) 12.118 V(TE)/ML 4.533
1 V/ML 1.184 PH(M) 10.518
2 V/ML 1.703 PH(M) 7.402
3 V/ML 2.036 PH(M) 4.823

0.25ML/DIV V(START)/ML 0.000 PH

2 3 4 5 6 7 8 9 10 11 12
| | | | | | | | | | | |

Kurath (Triplicate)

100μL of 00-1701 (3)

5/9/00

mg

ROUTINE # 101
9 PH(INIT) 12.151 V(TE)/ML 4.098
1 V/ML 1.169 PH(M) 10.473
2 V/ML 1.700 PH(M) 7.313
3 V/ML 2.023 PH(M) 4.839

DATE

NAME

BRINKMAN CAT # 2025015-1

Kurath MS

100% of 00-1701

+ 2ml of 0.1186 M NaOH

rg3 5/9/00

ROUTINE # 101
10 PH(INIT) 11.810 V(TE)/ML 4.694
1 V/ML 2.214 PH(M) 10.251
2 V/ML 2.785 PH(M) 7.449
3 V/ML 3.194 PH(M) 4.740

DATE

NAME



... Putting Technology To Work

Project No. 29953

Internal Distribution
File/LB

Date May 9, 2000
To D. E. Kurath
From S. K. Fiskum *S.K. Fiskum*
Subject Radiochemical Analyses for AP-101 Composite
ASR 5778

A sample of the composite from tank AP-101 (00-1701) was analyzed in triplicate for total alpha, total beta, gamma energy analysis, ^3H , uranium, ^{90}Sr , ^{99}Tc , Pu, and Am/Cm. The attached reports list measured analyte activities in the original AP101 material in units of $\mu\text{Ci/ml}$ except for uranium, which is reported in $\mu\text{g/ml}$. The reported errors ($1-\sigma$) represent the total propagated error including counting, dilution, yield, and calibration errors, as appropriate. Laboratory and process blank values given with each analysis are the best indicators of the method detection limits, taking into account the actual sample sizes and counting times used for each analysis.

Gamma Spectrometry

Triplicate 100- μL aliquots of AP101 original material were prepared into 2-mL geometries with deionized water (no acid digestion was applied). The small aliquot size was necessary to reduce the detector dead time attributed to the high ^{137}Cs activity. The samples were counted and analyzed directly by gamma energy analysis (GEA) according to procedure PNL-ALO-450. Because no sample manipulation was performed other than simple aliquoting and diluting, no preparation blank was prepared. In order to meet the required detection limits, the samples were counted for 14 hours each. Only ^{137}Cs , ^{134}Cs , and ^{60}Co could be identified in the gamma spectra. The triplicate concentrations of ^{137}Cs , ^{134}Cs , and ^{60}Co agreed to within $\pm 3\%$, well within the $< 15\%$ RSD acceptance criteria. The other requested analyte concentrations were below the minimum reporting limits requested.

Pertechnetate ^{99}Tc

The radiochemical ^{99}Tc determination was requested to measure only Tc in the +7 oxidation state (pertechnetate). To this end, all sample manipulations had to be non-oxidizing so as not to alter the original Tc oxidation state. Small aliquots from the as-received material (no digestion) were taken for analysis according to procedure PNL-ALO-432. This procedure normally requires the use of sodium dichromate addition to oxidize the Tc to the +7 oxidation state. The sodium dichromate addition was omitted and the procedure otherwise was performed as written. The separated fractions were then counted according to RPG-CMC-408. One sample was also counted by gamma spectrometry (according to PNL-ALO-450) to verify the absence of ^{137}Cs . The LCS and matrix spike were spiked with pertechnetate and their recoveries were 99% and 84%, respectively, well

within project requirements. The RSD of the triplicate samples was 8%, again well within the project-required < 15% RSD.

Acid Digestion

Triplicate 0.5-mL aliquots of AP101 were digested with nitric acid according to PNL-ALO-106. Heating and nitric acid additions continued until all organic/nitric reactions had visibly ceased. The samples were then brought to a 10-mL volume with 2M HNO₃. A preparation blank was run side by side with the samples. These digestions were used for the subsequent analyses of total alpha, total beta, ²³⁹⁺²⁴⁰Pu, ²³⁸Pu, ²⁴¹Am, ²⁴²Cm, ²⁴³⁺²⁴⁴Cm, total U, and ⁹⁰Sr. A laboratory control sample (LCS) was not available for radiochemical analyses. Specific reagent spikes or blank spikes were prepared subsequent to digestion and were used as LCSs.

Total Alpha

The total alpha activity was determined by direct-plating small aliquots of the acid-digested samples onto planchets according to RPG-CMC-4001. The samples were then counted on Ludlum detectors according to RPG-CMC-408. The sample activities resulted in a 9% RSD, well within the < 15% RSD acceptance criteria. Alpha activity was not found in the preparation blank. The LCS ²³⁹Pu recovery was 99% and the matrix spike ²³⁹Pu recovery was only 55%. The low matrix spike recovery is attributed to the solids loading on the planchet from the sample matrix salts that attenuate/absorb the alpha emissions. This is a physical problem with the sample as it is presented to the detector and does not indicate the analysis is out of control. What this does indicate is that the analytical method is probably biased low by as much as 45% for this matrix. A better indication of the total alpha activity is given by the summation of the alpha emitters (²³⁹⁺²⁴⁰Pu, ²³⁸Pu, ²⁴¹Am, ²⁴³⁺²⁴⁴Cm and ²⁴²Cm), shown in the last column of the data report. These alpha-emitter summations range from 18% to 39% higher than the total alpha activity found by direct plating. The RSD for the summation method is 1%.

Total Beta

The total beta activity was determined by directly plating small aliquots of the acid-digested samples onto planchets according to RPG-CMC-4001. The samples were then counted on a low-background alpha/beta gas-flow proportional counter according to RPG-CMC-408. The detectors were calibrated for beta activity relative to ⁹⁰Sr/⁹⁰Y. The beta energy of ¹³⁷Cs is similar to that of ⁹⁰Sr and will have a counting efficiency similar to that of pure ⁹⁰Sr. The total beta analyses compared to the ¹³⁷Cs determinations shows that virtually all of the beta activity is due to ¹³⁷Cs and the two values are in excellent agreement. The precision, measured by the RSD, is 5%, within experimental error. The LCS and matrix spike showed excellent ⁹⁰Sr/⁹⁰Y recoveries at 100% and 103%, respectively.

Plutonium, Americium, and Curium

The Pu and Am/Cm separations were performed according to PNL-ALO-417; the separated fractions were precipitation plated according to PNL-ALO-496; and the samples were counted by alpha spectrometry according to PNL-ALO-422. Plutonium recovery was traced with ²⁴²Pu. The curium is known to follow the americium and both these isotopes were traced with ²⁴³Am.

Plutonium radiochemical yields were excellent at > 90%. Radiochemical yields for Am ranged from 70% to 86%. In both cases, ample counts were obtained to define sample activities. Neither Pu, Am nor Cm were detected in the preparation blank. The LCS and matrix spike for Pu resulted in 98% and 101% yield-corrected recovery. This indicates the chemistry and analyses were not biased. The LCS and matrix spike for the Am (and Cm) fractions resulted in 94% and 91% yield-corrected recovery. The $^{239+240}\text{Pu}$ analyses resulted in 2% RSD, for ^{238}Pu the RSD was 5%. The ^{241}Am resulted in 2% RSD, the $^{243+244}\text{Cm}$ resulted in 29% RSD. This latter high RSD reflects the much higher uncertainty associated with the analytical results, which are approximately 5 orders of magnitude less than the requested detection limit.

Strontium-90

The Sr separation was performed according to PNL-ALO-476 and radiochemical yields were traced with ^{85}Sr . The separated fractions were then beta-counted according to RPG-CMC-408 and gamma counted according to PNL-ALO-450 (for ^{85}Sr determination and ^{137}Cs impurity assessment). Two of the separated fractions contained a small amount of ^{137}Cs and a correction to the beta count rate was applied for these samples. Strontium-90 was not found in the preparation blank. The RSD is 0.3%, indicative of excellent precision. The LCS and matrix spike recoveries were 92% and 99%, respectively, indicating good accuracy.

Total Uranium

Total uranium was measured in triplicate aliquots taken from the acid digestion according to procedure PNNL-ALO-4014 using Kinetic Phosphorescence Analysis (KPA). The triplicate results showed excellent agreement with a RSD of 1%. A small amount of uranium was detected in the acid digestion process blank; however, the level was a factor of 10,000 lower than the uranium seen in the samples. A blank prepared at the time of the analysis did not detect any uranium. Uranium standards analyzed before and after the samples agreed within 3% of the known values.

Tritium

Tritium was analyzed by distillation of direct sample material using procedure PNL-ALO-418 followed by liquid scintillation counting according to procedure PNL-ALO-474. The first distillation showed the presence of higher-energy beta activity, most likely due to ^{137}Cs . A second distillation succeeded in removing most of this contamination; however, two of the triplicate samples required subtraction of weak beta contamination using the ratio of the counts in the tritium energy region to the counts at higher energies, as determined from the first counting results. The triplicate results showed good agreement with a RSD of 5%. A blank spike showed good recovery at 96%. No tritium was detected in a blank prepared at the time of analysis in the laboratory.

Battelle / Pacific Northwest National Laboratory
 Radiochemical Processing Laboratory / 325 Building
 Radiochemical Processing Group

Cognizant Scientist: Richard T. R. Date: 5/2/00
 Technical Reviewer: L. Greenwood Date: 5/2/00

Client: Kurath
 MT&E Instrument: GEA Detector G
 Reference Date: N.A.

Gamma Energy Analysis (Procedure PNL-ALO-450):

Sample ID ALO Number	Measured Activity in $\mu\text{Ci/ml}$						
	Co-60 1 σ Unc.	Ru/Rh-106 1 σ Unc.	Sb-125 1 σ Unc.	Cs-134 1 σ Unc.	Cs-137 1 σ Unc.	Eu-152 1 σ Unc.	Eu-154 1 σ Unc.
AP-101 Composite 00-01701	3.22E-3 7%	<9E-2	<6E-2	4.77E-2 3%	1.44E+2 2%	<2E-3	<4E-2
AP-101 Composite Dup. 00-01701 Dup	3.40E-3 7%	<9E-2	<6E-2	4.81E-2 3%	1.44E+2 2%	<3E-3	<4E-2
AP-101 Composite Trip. 00-01701 Trip	3.24E-3 7%	<9E-2	<6E-2	4.62E-2 3%	1.45E+2 2%	<2E-3	<4E-2
RSD	3.0%			2.1%	0.7%		

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

00-1701

5/8/2000

Client: Kurath

Cognizant Scientist: S.K. Shukla

Date:

5/8/00

Concur: L.R. Green

Date:

5/8/00

Measured Activities (uCi/ml) with 1-sigma error

ALO ID Client ID	Total		Pu-239+		Pu-238		Pu-236		Am-241		Cm-243+		Cm-242		Alpha*	
	Alpha Error %		Pu-240 Error %		Pu-238 Error %		Pu-236 Error %		Am-241 Error %		Cm-244 Error %		Error %		Sum	
00-1701 PB AP-101 Comp	<7.E-5		<2.E-7		<2.E-7		<2.E-7		<5.E-7		<8.E-8		<8.E-8			
00-1701 AP-101 Comp	2.55E-4 14%		1.24E-4 4%		1.39E-5 6%		<9.E-8		1.73E-4 5%		1.34E-6 19%		<1.E-7		3.12E-4	
00-1701 Dup AP-101 Comp	2.22E-4 14%		1.28E-4 4%		1.49E-5 6%		<9.E-8		1.65E-4 5%		7.40E-7 24%		<9.E-8		3.09E-4	
00-1701 T AP-101 Comp	2.65E-4 14%		1.27E-4 4%		1.54E-5 6%		<2.E-7		1.70E-4 5%		1.02E-6 21%		<2.E-7		3.13E-4	
RSD	9%		2%		5%				2%		29%				1%	
Matrix Spike	55%*		101%						91%							
Blank Spike	99%		98%						94%							
Blank	<8.E-5															

*The low matrix spike recovery is attributed to plated solids causing absorption/attenuation of alpha emissions. The ramifications of this are the sample activities are probably similarly affected. The sum of the alpha emitters in the last column is thus the best estimate of the total alpha activity.

The total alpha, total beta, Pu, Am, Sr, and U analyses were performed on sample aliquots prepared according to PNL-ALO-106. This is a digestion procedure using nitric acid and heating that destroys organic material.

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

00-1701

5/4/2000

Client : Kurath

Cognizant Scientist:

L R Greenwood

Date :

5/4/00

Concur :

V K Hoken

Date :

5/8/00

Measured Activities (uCi/ml) with 1-sigma error

ALO ID Client ID	Beta Error %	Tc-99 as pertechnetate Error %	Sr-90 Error %
00-1701 PB AP-101 Comp	8.57E-5 31%	<2.E-5	<2.E-4
00-1701 AP-101 Comp	1.46E+2 4%	4.71E-2 4%	8.71E-2 3%
00-1701Dup AP-101 Comp	1.40E+2 4%	4.34E-2 4%	8.77E-2 3%
00-1701 T AP-101 Comp	1.54E+2 4%	5.10E-2 4%	8.75E-2 3%
Matrix Spike	103%	84%	99%
Blank Spike	100%	99%	92%
Method detection limit	2.E-1**	6.E-5	<6.E-4

**Calculated with respect to specific sample-counting conditions, i.e., small sample size, short count time. A larger preparation blank sample aliquot was taken and longer counting times applied, resulting in a lower detection limit for the preparation blank.

The total alpha, total beta, Pu, Am, Sr, and U analyses were performed on sample aliquots prepared according to PNL-ALO-106. This is a digestion procedure using nitric acid and heating that destroys organic material.

Tc-99 determination was performed on an aliquot with no acid digestion, no sample oxidation. Thus, the Tc-99 represents the pertechnetate form.

Client : Kurath

Cognizant Scientist:

L. R. Curran

Date :

5/3/00

Concur :

D. K. Aiken

Date :

5/3/00

ALO ID Client ID	Uranium ug/ml Error %	+/-%
00-1701 PB AP-101 Comp	5.41E-3	3%
00-1701 AP-101 Comp	5.17E+1	3%
00-1701Dup AP-101 Comp	5.12E+1	3%
00-1701 T AP-101 Comp	5.03E+1	3%
RSD	1%	
Blank	<1.E-5	

	Standard	Measured	Ratio
Prerun Standard 1	1.00E-2	9.92E-3	0.992
Prerun Standard 2	1.00E-1	1.02E-1	1.020
Prerun Standard 3	1.00E+0	1.09E+0	1.089
Post Standard 1	1.00E-2	9.71E-3	0.971
Post Standard 2	1.00E-3	1.01E-3	1.010

The total alpha, total beta, Pu, Am, Sr, and U analyses were performed on sample aliquots prepared according to PNL-ALO-106. This is a digestion procedure using nitric acid and heating that destroys organic material.

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

00-1701
5/9/2000

Client : Kurath

Cognizant Scientist:

C. Soderqvist

Date :

5-9-00

Concur :

L. H. Jensen

Date :

5-9-00

Measured Activities (uCi/ml) with 1-sigma error

ALO ID Client ID	Tritium Error %
00-1701	5.05E-3
AP-101 Comp	4%
00-1701Dup	4.84E-3
AP-101 Comp	3%
00-1701 T	4.60E-3
AP-101 Comp	3%
RSD	5%
Blank Spike	96%
Blank	< 2.E-4

Note: Tritium was determined by liquid scintillation counting following two successive distillations of direct sample material. Weak contamination was observed in the beta spectra of the first two results reported above. This contamination was subtracted out of the tritium results reported above. (We used data from a previous attempt at this analysis to accurately calculate the amount of contamination to subtract.) The third result above did not show any significant contamination.



Battelle

... Putting Technology To Work

Project No. 29953

Internal Distribution
File/LB

Date May 12, 2000
To D. E. Kurath
From L. R. Greenwood *LR Greenwood*
Subject C-14 Analyses for AP-101 Composite ASR 5778

Direct samples of the composite from tank AP-101 (00-1701) were analyzed in triplicate for C-14 following procedure PNL-ALO-482. The samples are combusted in a Coulometrics Carbon Analyzer Furnace causing oxidation of all carbon species present to CO₂. A natural carbon compound is added as a carrier and all of the CO₂ released is collected in a sodium hydroxide trap. The trap solution is then counted by liquid scintillation counting according to procedure PNL-ALO-474. The triplicate results showed good agreement with a relative standard deviation of 5%. The blank and sample spikes showed good recovery at 97% and 96%, respectively. Due to the inadvertent spiking of a system blank prior to the analysis, some carryover was seen in the prior blanks. However, a system blank analyzed following the analyses did not show any contamination.

Client : Kurath

Cognizant Scientist: L R Heenan

Date: 5/12/00

Concur: Wanda K. Fick

Date: 5/12/00

Procedures PNL-ALO-482/474

Measured Activities (uCi/ml) with 1-sigma error

ALO ID	C-14
Client ID	Error %
00-1701	2.56E-4
AP-101 Comp	8%
00-1701Dup	2.55E-4
AP-101 Comp	8%
00-1701 T	2.77E-4
AP-101 Comp	8%
RSD	5%
Blank	< 4.E-5
Sample Spike	96%
Standard	97%



... Putting Technology To Work

Project No. 29953

Internal Distribution
File/LB

Date May 18, 2000
To D. E. Kurath
From L. R. Greenwood *LRG*
Subject Se-79 Analyses for AP-101 Composite ASR 5778

Direct samples of the composite from tank AP-101 (00-1701) were analyzed in triplicate for Se-79 following procedure PNL-ALO-440. Each sample was spiked with 20 mg Se carrier in solution to be used for yield correction. Anion and cation exchange was used to remove most radiochemical interferences. Selenium was distilled as selenium bromide and then reduced to elemental form. The chemical yield was determined gravimetrically by weighing the recovered elemental selenium. The selenium is then dissolved and the Se-79 activity was determined by liquid scintillation counting according to procedure PNL-ALO-474. The liquid scintillation spectra did not show clear evidence for a Se-79 peak and there appears to be some weak beta contamination evident in the higher energy region of the beta spectrum. Hence, the results probably have a high bias. We did not rerun or attempt to further correct the data since the measured Se-79 activities are below the requested MRQ value of $9.0E-5$ uCi/ml. The nominal MDL was $2.E-5$ uCi/ml. The sample activity averaged $5.8E-5$ uCi/ml with an RSD of 18%. The RSD acceptance criteria of $< 15\%$ was exceeded, although the measurement uncertainties were as high as 11% on one sample and the sample activity is only a factor of three higher than the MDL. Since Se-79 is not available as a standard, C-14 was used to calibrate the detector since C-14 has nearly the same beta energy as Se-79. Due to the lack of a Se-79 standard, blank and sample spikes are not possible. The sample carrier recoveries were low at 27% to 63% and the blank recovery was 68%. Sample activities were yield-corrected. A process blank did not show any evidence of contamination.

Client: Kurath

Cognizant Scientist:

J.R. Greenwood

Date:

5/18/00

Concur:

D.K. Fisk

Date:

5/18/00

Procedures PNL-ALO-440/474

Measured Activities (uCi/ml) with 1-sigma error

ALO ID	Se-79
Client ID	Error %
00-1701	5.56E-5
AP-101 Comp	8%
00-1701Dup	6.97E-5
AP-101 Comp	5%
00-1701 T	4.93E-5
AP-101 Comp	11%
RSD	18%
Blank	< 3.E-6
Blank Spike Recovery	68%

Note: The beta energy spectra did not show clear evidence for a Se-79 peak and there appears to be some beta contamination at higher beta energies. Hence, the results probably have a high bias. All measurements are below the requested MRQ value of 9.0E-5 uCi/ml.

**Battelle**

Pacific Northwest Laboratories

Internal Distribution

Date May 10, 2000

329 File
Mike Urie

To Dean Kurath

From Tom Farmer *Quill Thomas Farmer*Subject ICPMS Analysis AP101 Samples
(ALO# 001701)

Pursuant to your request, the 6 samples that you submitted for analysis were analyzed on our radioactively-contained ICPMS for the selected analytes; semiquantitative analysis was necessary on certain isotopes for which a standard was not available (see below). The concentration results for the isotopes of interest are displayed on the attached spreadsheets.

Dilutions of Isotope Products standards for ^{129}I , ^{237}Np and ^{239}Pu , an Amersham ^{99}Tc standard, an NIST uranium standard (4321b), an ^{127}I standard, made from Fisher potassium iodide (LOT# 35260), and CPI single element standards for As, B, Be, Ce, Co, Cs, Eu, Li, Mo, Pr, Rb, Sb, Se, Ta, Te, Th, Ti, V, W and Sn, were used to generate the calibration curves. Independent standards of each analyte were used as the continuing calibration verification (CCV) standards. The 1% high-purity nitric acid solution used to dilute the standards and samples was used as a reagent blank. The results are reported in μg analyte /ml (ppm), or μCi analyte /ml of original sample material.

In general, the results for the quality control samples (i.e., calibration verification standards, duplicates, and matrix spikes) are well within acceptance criteria. One of the CCV results for tellurium and tantalum and the spike recovery for thallium, were outside the acceptance window (<20% for the CCV and <30% for the Spike). Tellurium and tantalum did have another CCV results that was acceptable. CCV results for Thallium were also acceptable. Also the the duplicate and triplicate for arsenic and the duplicate for molybdenum were outside the the acceptance window (<15%).

The ^{99}Tc values reported assume that the Ru present is exclusively fission-product Ru, and therefore does not have an isotope at m/z 99; i.e., everything observed at m/z 99 is due to ^{99}Tc . From the appearance of the Ru isotopic abundance, this appears to be a reasonable assumption; the fingerprint exhibited is obviously not natural.

Values for the following isotopes were obtained using responses from related isotopes: ^{126}Sn (obtained from ^{118}Sn), ^{231}Pa (obtained from ^{232}Th), and ^{240}Pu , ^{241}AMU , ^{242}AMU , ^{243}AMU (obtained from ^{239}Pu). Because standards were not used and the concentrations of the isotopes were determined indirectly, these results should be considered semiquantitative.

If you have any questions regarding this analysis, please give me a call at 372-0700 or James Bramson at 376-0624.

AP101 Analysis

May 17, 2000

OTF

JPB
5/17/00
Pg 1 of 8

Sample ID	Client ID	Lithium		Beryllium		Boron	
		MDL µg/ml	Analyzed µg/ml ± 1SD	MDL µg/ml	Analyzed µg/ml ± 1SD	MDL µg/ml	Analyzed µg/ml ± 1SD
00-001701PB	PROCESS BLANK	<2.3	< 0.064	<2.3	0.012 ± 0.0024	<2.3	6.42 ± 0.16
00-001701BS	BLANK SPIKE	<2.3	< 0.72	<2.3	0.12 ± 0.016	<2.3	118 ± 1.2
00-001701	AP-101-COMP	<2.3	0.323 ± 0.004	<2.3	1.32 ± 0.03	<2.3	15.5 ± 0.1
00-001701D	AP-101-COMP	<2.3	0.329 ± 0.002	<2.3	1.28 ± 0.01	<2.3	15.3 ± 0.1
00-001701T	AP-101-COMP	<2.3	0.367 ± 0.003	<2.3	1.35 ± 0.01	<2.3	17.2 ± 0.1
00-001701+spike Spike Recovery	AP-101-COMP	<2.3	1.95 ± 0.01 123%	<2.3	2.71 ± 0.01 105%	<2.3	17.6 ± 0.1 105%
00-001701MS	AP-101-COMP	<2.3	0.360 ± 0.002	<2.3	1.39 ± 0.01	<2.3	18.6 ± 0.2
Blank and CCV results are reported in ng/ml (ppb)							
1%HNO3			< 0.27		< 0.018		< 0.71
1%HNO3			< 0.28		0.139 ± 0.012		1.0 ± 0.6
1ppb Multi			0.837 ± 0.018		0.977 ± 0.018		0.81 ± 0.08
10ppb Multi			11 ± 2		11 ± 2		11 ± 2

AP101 Analysis

May 17, 2000 (revised May 18, 2000)

OTF 18 May 00

2813

Sample ID	Client ID	MDL µg/ml	Vanadium Analyzed µg/ml	MDL µg/ml	±	1SD	MDL µg/ml	Cobalt Analyzed µg/ml	MDL µg/ml	±	1SD	MDL µg/ml	Arsenic Analyzed µg/ml	MDL µg/ml	±	1SD
00-001701PB	PROCESS BLANK	<2.3	< 0.79	<2.3			<2.3	0.014	<2.3	±	0.003	<2.3	0.13	<2.3	±	0.004
00-001701BS Spike Recovery	BLANK SPIKE	<2.3	< 8.8	<2.3			<2.3	< 0.139	<2.3			<2.3	373 117%	<2.3	±	1
00-001701	AP-101-COMP	<2.3	< 0.77	<2.3			<2.3	0.356	<2.3	±	0.005	<2.3	1.46	<2.3	±	0.03
00-001701D	AP-101-COMP	<2.3	< 0.75	<2.3			<2.3	0.331	<2.3	±	0.007	<2.3	1.15	<2.3	±	0.044
00-001701T	AP-101-COMP	<2.3	< 0.72	<2.3			<2.3	0.369	<2.3	±	0.001	<2.3	1.18	<2.3	±	0.017
00-001701+spike Spike Recovery	AP-101-COMP	<2.3	2.23 110%	<2.3	±	0.01	<2.3	1.75 108%	<2.3	±	0.01	<2.3	2.97 109%	<2.3	±	0.02
00-001701MS Spike Recovery	AP-101-COMP	<2.3	< 0.66	<2.3			<2.3	0.368	<2.3	±	0.003	<2.3	29.7 120%	<2.3	±	0.05
Blank and CCV results are reported in ng/ml (ppb)																
1%HNO3			< 3.3					< 0.053					< 0.27			
1%HNO3			< 3.4					0.132			±	0.009	< 0.28			
1ppb Multi			0.970		±	0.043		0.979			±	0.009	0.942		±	0.035
10ppb Multi			12		±	2		11			±	2	11		±	2

AP101 Analysis

May 17, 2000 (revised May 18, 2000)

OTF 18 May 00

Sample ID	Client ID	Selenium		Rubidium		Molybdenum	
		MDL µg/ml	Analyzed µg/ml ± 1SD	MDL µg/ml	Analyzed µg/ml ± 1SD	MDL µg/ml	Analyzed µg/ml ± 1SD
00-001701PB	PROCESS BLANK	<2.3	< 2.3	<2.3	0.017 ± 0.004	<2.3	< 0.013
00-001701BS	BLANK SPIKE	<2.3	65.1 102% ± 0.14	<2.3	0.387 ± 0.006	<2.3	2.76 ± 0.32
Spike Recovery 00-001701	AP-101-COMP	<2.3	< 2.3	<2.3	3.92 ± 0.01	<2.3	15.9 ± 0.2
00-001701D	AP-101-COMP	<2.3	< 2.3	<2.3	3.78 ± 0.02	<2.3	13.4 ± 0.1
00-001701T	AP-101-COMP	<2.3	< 2.3	<2.3	4.22 ± 0.03	<2.3	14.1 ± 0.1
00-001701+spike Spike Recovery	AP-101-COMP	<2.3	< 2.3	<2.3	5.46 106% ± 0.02	<2.3	18.0 105% ± 0.2
00-001701MS	AP-101-COMP	<2.3	5.8 118% ± 0.015	<2.3	4.13 ± 0.01	<2.3	18.2 ± 0.3
Spike Recovery							

Blank and CCV results are reported in ng/ml (ppb)

1%HNO3	< 0.45	< 0.050	< 0.057
1%HNO3	< 0.45	0.136 ± 0.004	0.288 ± 0.063
1ppb Multi	< 2.3	1.02 ± 0.02	0.920 ± 0.015
10ppb Multi	12 ± 3	11 ± 2	11 ± 2

5/18/00
3 of 13

AP101 Analysis **OTF**

May 17, 2000

J.P. Brown
5/17/00
PS 5 of 13

Sample ID	Client ID	*Tin 126 MDL μCi/ml	*Tin 126 Analyzed μCi/ml	Iodine 127 Analyzed μg/ml	Iodine 127 MDL μCi/ml	Iodine 129 Analyzed μCi/ml	Iodine 129 MDL μCi/ml	± 1SD	± 1SD
00-001701PB	PROCESS BLANK	<6.0E-03	< 1.3E-03	<0.013	<1.8E-05	< 2.5E-06	<1.8E-05	± 0.04	± 5.2E-06
00-001701BS	BLANK SPIKE	<6.0E-03	< 1.0E-02	<0.18	<1.8E-05	< 3.4E-05	<1.8E-05	± 0.10	± 3.9E-06
00-001701	AP-101-COMP	<6.0E-03	< 8.6E-04	2.42	<1.8E-05	7.72E-05	<1.8E-05	± 0.08	± 2.7E-06
00-001701D	AP-101-COMP	<6.0E-03	< 8.6E-04	2.49	<1.8E-05	7.73E-05	<1.8E-05	± 0.17	± 1.0E-05
00-001701T	AP-101-COMP	<6.0E-03	< 8.2E-04	2.73	<1.8E-05	8.71E-05	<1.8E-05	± 0.04	± 2.3E-06
00-001701+spike Spike Recovery	AP-101-COMP	<6.0E-03	< 8.2E-04	3.48 94%	<1.8E-05	1.20E-04 99%	<1.8E-05	± 0.04	± 2.3E-06
00-001701MS	AP-101-COMP	<6.0E-03	< 7.4E-04	2.93	<1.8E-05	9.10E-05	<1.8E-05	± 0.04	± 2.3E-06
Blank and CCV results are reported in ng/ml (ppb)									
1%HNO3		< 0.13		0.423		< 0.061		± 0.013	
1%HNO3		< 0.13		0.160		< 0.120		± 0.012	
0.502ppb I-127				0.551				± 0.024	
5.02ppb I-127				5.04				± 0.28	
0.1ppb I-129						0.0981		± 0.0133	
1ppb I-129						1.12		± 0.01	

*Calculated from response of different isotope. Should be considered semiquantitative.

AP101 Analysis

May 17, 2000

OTF

Sample ID	Client ID	MDL μg/ml	Cesium Analyzed μg/ml	±	1SD	MDL μg/ml	Cerium Analyzed μg/ml	±	1SD	MDL μg/ml	Praseodymium Analyzed μg/ml	±	1SD
00-001701PB	PROCESS BLANK	<1.5	0.0136	±	0.0006	<2.3	<0.079			<2.3	<0.018		
00-001701BS	BLANK SPIKE	<1.5	0.50	±	0.04	<2.3	<0.88			<2.3	<0.203		
00-001701	AP-101-COMP	<1.5	4.96	±	0.02	<2.3	<0.077			<2.3	<0.018		
00-001701D	AP-101-COMP	<1.5	4.90	±	0.05	<2.3	<0.075			<2.3	<0.017		
00-001701T	AP-101-COMP	<1.5	5.40	±	0.04	<2.3	<0.072			<2.3	<0.017		
00-001701+spike Spike Recovery	AP-101-COMP	<1.5	6.70 108%	±	0.06	<2.3	1.57 117%	±	0.01	<2.3	1.51 118%	±	0.02
00-001701MS	AP-101-COMP	<1.5	5.32	±	0.06	<2.3	<0.066			<2.3	<0.015		
Blank and CCV results are reported in ng/ml (ppb)													
1%HNO3			<0.018				<0.33				<0.077		± 0.011
1%HNO3			0.138	±	0.002		<0.34				0.139		± 0.032
1ppb Multi			1.10	±	0.03		0.928	±	0.031		0.963	±	± 2
10ppb Multi			11	±	2		11	±	2		11	±	2

Handwritten notes:
JMA
5/11/00
pg 6 of 13

AP101 Analysis

May 17, 2000

JPB
5/17/00
pg 7 of

Sample ID	Client ID	MDL µg/ml	Europium Analyzed µg/ml	±	1SD	MDL µg/ml	Tantalum Analyzed µg/ml	±	1SD	MDL µg/ml	Tungsten Analyzed µg/ml	±	1SD
00-001701PB	PROCESS BLANK	<60	< 0.017			<2.3	< 0.087			<2.3	< 0.089		
00-001701BS	BLANK SPIKE	<60	0.424	±	0.014	<2.3	< 1.12			<2.3	< 1.04		
00-001701	AP-101-COMP	<60	< 0.016			<2.3	< 0.087			<2.3	28.5	±	1.2
00-001701D	AP-101-COMP	<60	< 0.016			<2.3	< 0.089			<2.3	28.4	±	0.7
00-001701T	AP-101-COMP	<60	< 0.015			<2.3	< 0.089			<2.3	28.9	±	1.3
00-001701+spike Spike Recovery	AP-101-COMP	<60	1.47 115%	±	0.01	<2.3	0.190 89%	±	0.002	<2.3	47.9 124%	±	1.7
00-001701MS	AP-101-COMP	<60	0.015	±	0.003	<2.3	< 0.084			<2.3	26.0	±	0.2
Blank and CCV results are reported in ng/ml (ppb)													
1%HNO3			< 0.071				< 0.37				< 0.092		
1%HNO3			0.140	±	0.007		< 0.34				0.87	±	0.22
1ppb Multi			0.961	±	0.019								
10ppb Multi			11	±	2								
1ppb/0.5ppb Multi							1.1	±	0.2				
0.5ppb Multi							0.717	±	0.030				

OTF

AP101 Analysis

May 17, 2000

8/15/00
5/17/00
pg 8 of 17

Sample ID	Client ID	MDL µg/ml	Thallium Analyzed µg/ml	±	1SD	MDL µCi/ml	*Protactinium 231 Analyzed µCi/ml	MDL µg/ml	Thorium Analyzed µg/ml	±	1SD
00-001701PB	PROCESS BLANK	<2.3	< 0.0055			<7.9E-05	< 1.0E-04	<2.3	< 0.0110		
00-001701BS	BLANK SPIKE	<2.3	310	±	8	<7.9E-05	< 1.3E-03	<2.3	< 0.14		
00-001701	AP-101-COMP	<2.3	0.0192	±	0.0010	<7.9E-05	< 1.0E-04	<2.3	< 0.011		
00-001701D	AP-101-COMP	<2.3	0.0168	±	0.0006	<7.9E-05	< 1.1E-04	<2.3	< 0.011		
00-001701T	AP-101-COMP	<2.3	0.0165	±	0.0013	<7.9E-05	< 1.1E-04	<2.3	< 0.011		
00-001701+spike Spike Recovery	AP-101-COMP	<2.3	0.203 139%	±	0.005	<7.9E-05	< 1.1E-04	<2.3	0.168 122%	±	0.001
00-001701MS	AP-101-COMP	<2.3	27.2	±	0.2	<7.9E-05	< 1.0E-04	<2.3	< 0.0107		
Blank and CCV results are reported in ng/ml (ppb)											
1%HNO3		< 0.023					< 0.0093		< 0.047		
1%HNO3		0.36		±	0.06		< 0.0087		< 0.043		
1ppb Multi											
10ppb Multi											
1ppb/0.5ppb Multi		0.493		±	0.008				0.558	±	0.032
0.5ppb Multi		0.500		±	0.036				0.497	±	0.013

*Calculated from response of different isotope. Should be considered semiquantitative.

45

AP101 Analysis

May 17, 2000

Sample ID	Client ID	Uranium 233		Uranium 234		Uranium 235	
		MDL μCi/ml	Analyzed μCi/ml ± 1SD	MDL μCi/ml	Analyzed μCi/ml ± 1SD	MDL μCi/ml	Analyzed μCi/ml ± 1SD
00-001701PB	PROCESS BLANK	<4.2E-04	< 8.8E-06	<1.2E-4	< 5.7E-06	<4.5E-08	< 6.8E-09
00-001701BS	BLANK SPIKE	<4.2E-04	< 8.8E-06	<1.2E-4	< 6.3E-05	<4.5E-08	< 4.6E-08
00-001701	AP-101-COMP	<4.2E-04	5.5E-05 ± 3.8E-06	<1.2E-4	2.7E-05 ± 5.7E-06	<4.5E-08	1.06E-06 ± 2.8E-09
00-001701D	AP-101-COMP	<4.2E-04	5.6E-05 ± 3.8E-06	<1.2E-4	2.4E-05 ± 4.1E-06	<4.5E-08	9.94E-07 ± 2.8E-09
00-001701T	AP-101-COMP	<4.2E-04	4.8E-05 ± 5.0E-06	<1.2E-4	2.1E-05 ± 2.4E-06	<4.5E-08	9.82E-07 ± 2.8E-09
00-001701+spike Spike Recovery	AP-101-COMP						
00-001701MS	AP-101-COMP	<4.2E-04	5.6E-05 ± 2.5E-05	<1.2E-4	2.2E-05 ± 8.2E-06	<4.5E-08	1.12E-06 ± 4.5E-08
Blank and CCV results are reported in ng/ml (ppb)							
1%HNO3			< 0.0007		< 0.0007		< 0.0007
1%HNO3							

AP101 Analysis

May 17, 2000

OTF

Sample ID	Client ID	Uranium 236		Uranium 238		Total Uranium Analyzed
		MDL μCi/ml	Analyzed μCi/ml ± 1SD	MDL μCi/ml	Analyzed μCi/ml ± 1SD	μg/ml ± 1SD
00-001701PB	PROCESS BLANK	<1.4E-06	< 5.9E-08	<7.2E-08	< 4.7E-08	< 0.14
00-001701BS	BLANK SPIKE	<1.4E-06	< 8.1E-08	<7.2E-08	< 5.3E-07	< 1.6
00-001701	AP-101-COMP	<1.4E-06	2.23E-06 ± 1.1E-07	<7.2E-08	1.96E-05 ± 8.8E-08	58.9 ± 2.1
00-001701D	AP-101-COMP	<1.4E-06	2.07E-06 ± 1.3E-07	<7.2E-08	1.76E-05 ± 8.8E-08	52.9 ± 1.8
00-001701T	AP-101-COMP	<1.4E-06	2.08E-06 ± 4.2E-08	<7.2E-08	1.80E-05 ± 8.8E-08	54.1 ± 1.7
00-001701+spike Spike Recovery	AP-101-COMP					91.3 ± 3.6 115%
00-001701MS	AP-101-COMP	<1.4E-06	2.39E-06 ± 1.7E-07	<7.2E-08	2.08E-05 ± 4.4E-08	62.3 ± 0.8
Blank and CCV results are reported in ng/ml (ppb)						
1%HNO3			< 0.0007		< 0.0007	< 0.15
1%HNO3						0.79 ± 0.33
20ppb U						19.5 ± 0.6
10/20ppb W/U						19.9 ± 0.6

5/17/00
pg 10 of 1

AP101 Analysis

May 17, 2000

OTF

Sample ID	Client ID	Neptunium 237			Plutonium 239		
		MDL μCi/ml	Analyzed μCi/ml	± 1SD	MDL μCi/ml	Analyzed μCi/ml	± 1SD
00-001701PB	PROCESS BLANK	<3.9E-05	< 5.3E-06		<3.0E-02	< 1.2E-03	
00-001701BS	BLANK SPIKE	<3.9E-05	< 6.9E-05		<3.0E-02	< 1.6E-02	
00-001701	AP-101-COMP	<3.9E-05	< 5.4E-06		<3.0E-02	< 1.2E-03	
00-001701D	AP-101-COMP	<3.9E-05	< 5.4E-06		<3.0E-02	< 1.2E-03	
00-001701T	AP-101-COMP	<3.9E-05	< 5.5E-06		<3.0E-02	< 1.2E-03	
00-001701+spike Spike Recovery	AP-101-COMP	<3.9E-05	1.12E-04 119%	± 3.0E-06	<3.0E-02	9.98E-03 110%	± 2.2E-04
00-001701MS	AP-101-COMP	<3.9E-05	< 5.2E-06		<3.0E-02	< 1.2E-03	
Blank and CCV results are reported in ng/ml (ppb)							
1%HNO3		< 0.032			< 0.082		
1%HNO3		< 0.030			< 0.077		
1ppb/0.5ppb Multi		0.508		± 0.031	0.445		± 0.022
0.5ppb Multi		0.498		± 0.027	0.506		± 0.023

5/17/00
pg 1 of 1

AP101 Analysis

May 17, 2000

OTF

Sample ID	Client ID	MDL μCi/ml	*Plutonium 240 Analyzed μCi/ml	*AMU 241 Analyzed μg/ml	*AMU 242 Analyzed μg/ml	*AMU 243 Analyzed μg/ml
00-001701PB	PROCESS BLANK	<3.0E-02	< 2.4E-03	< 0.011	< 0.010	< 0.0069
00-001701BS	BLANK SPIKE	<3.0E-02	< 3.1E-02	< 0.14	< 0.13	< 0.090
00-001701	AP-101-COMP	<3.0E-02	< 2.4E-03	< 0.011	< 0.010	< 0.0071
00-001701D	AP-101-COMP	<3.0E-02	< 2.4E-03	< 0.011	< 0.010	< 0.0072
00-001701T	AP-101-COMP	<3.0E-02	< 2.4E-03	< 0.011	< 0.010	< 0.0072
00-001701+spike Spike Recovery	AP-101-COMP	<3.0E-02	< 2.4E-03	< 0.011	< 0.010	< 0.0071
00-001701MS	AP-101-COMP	<3.0E-02	< 2.3E-03	< 0.010	< 0.0099	< 0.0068
Blank and CCV results are reported in ng/ml (ppb)						
1%HNO3		< 0.044		< 0.045	< 0.043	< 0.029
1%HNO3		< 0.041		< 0.042	< 0.040	< 0.028

*Calculated from response of different isotope. Should be considered semiquantitative.

Handwritten notes:
7/10
pg 12 of 11

JPB
5/10/00

AP101 Analysis

May 10, 2000

Sample ID	Client ID		U-233 Abundance	U-234 Abundance	U-235 Abundance	U-236 Abundance	U-238 Abundance
00-001701PB	PROCESS BLANK	Average	-4.24E-03	9.16E-03	1.20E-04	2.35E-03	9.88E-01
		stdev	9.56E-03	6.69E-03	6.77E-03	1.07E-02	2.18E-01
		%SD	-225.2%	73.1%	5657.7%	455.9%	22.0%
00-001701BS	BLANK SPIKE	Average	-6.68E-05	6.17E-03	1.31E-02	7.65E-04	9.80E-01
		stdev	1.13E-03	2.69E-03	9.78E-03	2.93E-03	2.66E-02
		%SD	-1691.0%	43.5%	74.7%	383.4%	2.7%
00-001701	AP-101-COMP	Average	9.75E-05	7.30E-05	8.32E-03	5.85E-04	9.91E-01
		stdev	6.79E-06	1.53E-05	9.00E-05	2.85E-05	5.45E-03
		%SD	7.0%	21.0%	1.1%	4.9%	0.5%
00-001701D	AP-101-COMP	Average	1.09E-04	7.44E-05	8.69E-03	6.04E-04	9.91E-01
		stdev	6.12E-06	9.09E-06	2.27E-04	3.48E-05	5.27E-03
		%SD	5.6%	12.2%	2.6%	5.8%	0.5%
00-001701T	AP-101-COMP	Average	9.15E-05	6.21E-05	8.41E-03	5.94E-04	9.91E-01
		stdev	8.74E-06	6.77E-06	2.62E-04	1.15E-05	4.22E-03
		%SD	9.5%	10.9%	3.1%	1.9%	0.4%
00-001701MS	AP-101-COMP	Average	9.32E-05	5.67E-05	8.31E-03	5.94E-04	9.91E-01
		stdev	3.21E-05	1.12E-05	3.12E-04	4.06E-05	2.48E-03
		%SD	34.5%	19.7%	3.8%	6.8%	0.3%
4321B		Average	6.25E-06	5.22E-05	7.08E-03	7.83E-06	9.93E-01
		stdev	1.12E-05	6.80E-06	2.80E-05	1.05E-05	1.32E-02
		%SD	178.8%	13.0%	0.4%	133.8%	1.3%
		True Value		5.29E-03	7.20E-01		9.93E+01
UO3O		Average	5.40E-06	1.91E-04	3.00E-02	1.97E-04	9.70E-01
		stdev	1.44E-05	1.53E-05	4.54E-04	6.85E-06	7.67E-03
		%SD	266%	8.0%	1.5%	3.5%	0.8%
		True Value		1.90E-04	3.05E-03	2.00E-04	9.69E-01

DATA REVIEW

Reviewed: *O.J. Jarman*

Date: *May 02* *13*

AP101 MDL's

Sample Number	Client Number	Li µg/ml	Be µg/ml	B µg/ml	V µg/ml	Co µg/ml	As µg/ml	Se µg/ml	Rb µg/ml	Mo µg/ml	Tc-99 µCi/ml	Sb µg/ml	Te µg/ml	Se-126 µCi/ml
00-001701	AP-101-COMP	0.063	0.004	0.16	0.007	0.012	0.063	2.3	0.011	0.013	2.6E-04	0.014	0.17	8.6E-04
00-001701D	AP-101-COMP	0.061	0.004	0.16	0.007	0.012	0.062	2.3	0.011	0.013	2.6E-04	0.014	0.16	8.6E-04
00-001701T	AP-101-COMP	0.059	0.004	0.15	0.007	0.011	0.059	2.3	0.011	0.012	2.6E-04	0.013	0.16	8.2E-04

Sample Number	Client Number	I-127 µg/ml	I-129 µCi/ml	Cs µg/ml	Ce µg/ml	Pr µg/ml	Eu µg/ml	Ta µg/ml	W µg/ml	Tl µg/ml	Pa-231 µCi/ml	Th µg/ml	U-233 µCi/ml	U-234 µCi/ml
00-001701	AP-101-COMP	0.037	6.7E-06	0.004	0.077	0.018	0.016	0.087	0.23	0.011	1.0E-04	0.011	8.8E-06	5.7E-06
00-001701D	AP-101-COMP	0.037	6.7E-06	0.004	0.075	0.017	0.016	0.089	0.22	0.011	1.1E-04	0.011	8.8E-06	5.7E-06
00-001701T	AP-101-COMP	0.033	6.0E-06	0.004	0.072	0.017	0.015	0.089	0.24	0.011	1.1E-04	0.011	8.8E-06	5.7E-06

Sample Number	Client Number	U-235 µCi/ml	U-236 µCi/ml	U µg/ml	Np-237 µCi/ml	Pu-239 µCi/ml	Pu-240 µCi/ml	AMU-241 µg/ml	AMU-242 µg/ml	AMU-243 µg/ml
00-001701	AP-101-COMP	6.8E-09	5.8E-08	0.090	5.4E-06	1.2E-03	2.4E-03	0.011	0.01	0.0071
00-001701D	AP-101-COMP	6.8E-09	5.8E-08	0.086	5.4E-06	1.2E-03	2.4E-03	0.011	0.01	0.0072
00-001701T	AP-101-COMP	6.8E-09	5.8E-08	0.094	5.5E-06	1.2E-03	2.4E-03	0.011	0.01	0.0072

WO/Project: W54906/29953
Client: D. Kurath

ACL Numbers: 00-01701
ASR Number 5778

Procedure: PNNL-ALO-131, "Mercury Digestion"
PNNL-ALO-201, "Mercury Analysis"

Analyst: J. J. Wagner

Digestion Date: May 10, 2000 Analysis Date: May 11, 2000

M&TE: Hg system (WD14126); Mettler AT400 Balance (360-06-01-029) See Chemical Measurement Center 98620 RIDS for Hg File for Calibration, Standards Preparations, and Maintenance Records.

Analyst: J. J. Wagner 5-15-00
Approval: [Signature] Date 5-15-00

Final Results:

The samples were analyzed by cold vapor atomic absorption spectrophotometry for inorganic mercury as specified in ASR 5778. Four aliquots of 0.2ml including one for matrix spike, was processed and diluted to a final volume of 25ml per procedure ALO-131. No additional dilution was performed. Concentration of all sample aliquots measured were near instrument detection limit and several times lower than MRQ (1.5 µg/ml) listed in Table 2, "Supernate Analyses Required by Contract Specification 7 and Opportunistic Analytes". The mercury concentration results are presented in the table below.

Lab ID	Solid Sample ID	Aqueous ml	Aqueous Dig Fctr	Aqueous Anal Fctr	Hg ug/ml	RPD (%)
00-01701-PB	Reagent Process Blank	0.20	125	1	0.026	
00-01701	AP-101 COMP	0.20	125	1	<0.025	
00-01701DUP	AP-101 COMP	0.20	125	1	<0.025	
00-01701TRI	AP-101 COMP	0.20	125	1	0.032	N/A

RPD = Relative Percent Difference (between sample and duplicate/replicate)

"Sample volume" used for the process blank is an average volume of the samples.

N/A = RPD is not calculated when results are less than 5 x IDL

Notes:

- 1) "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- 2) The low calibration standard is defined as the estimated detection limit (IDL) for the reported results and assumes non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested.
- 3) Routine precision and bias is typically $\pm 15\%$ or better for non-complex aqueous samples that are free of interference.

Q.C. Comments:

Following are results of quality control checks performed during Hg analyses. In general, quality control checks met the requirements of the governing QA Plan.

Working Blank Spike/Process Blank Spike: Process Blank Spike recovery is 100%, well within the acceptance criteria of 80% to 120%.

Matrix Spiked Sample: A matrix spike was prepared for the samples submitted under this ASR. Recovery of the matrix spike is 97%, well within the acceptance criteria of 75% to 125%.

Duplicate/replicates: RPD is not calculated since all replicate results are less than 5 x IDL.

Laboratory Control Sample (liquid): Sample recovery of mercury in SRM-1641d (certified by NIST to contain $1.60 \pm 0.018 \mu\text{g/ml}$) was recovered within acceptance criteria of 75% to 125%.

System Blank/Processing Blanks: A system blank was process during the analysis of the sample. The concentration measured was within about two times detection limit or less. Samples were about the same concentration as the process blank.

Quality Control Calibration Verification Check Standards: Six mid-range verification standards were analyzed throughout the analysis run. All were within the acceptance criteria of 80% to 120% recovery for the verification standard.



Battelle

... Putting Technology To Work

Project No. 29953

Internal Distribution
File/LB

Date May 18, 2000

To D. E. Kurath

From L. R. Greenwood *LRG*

Subject NH-3 Analyses for AP-101 Composite ASR 5778

Ammonia was measured in triplicate sample aliquots of the composite from tank AP-101 (00-1701) using an ion selective electrode according to procedure PNL-ALO-226. The ammonia probe was calibrated using five standards spanning ammonia concentrations from $1.0\text{E-}2$ to $1.0\text{E-}6$ moles/liter. The method of standard additions was used to determine the ammonia concentrations by first taking a direct reading and then adding a known standard to each sample. The triplicate concentrations are in good agreement with an RSD of 6%. The method detection limit was estimated at 0.2 ug/ml , well below the requested MRQ value of 140 ug/ml .

Client : Kurath

Cognizant Scientist:

J. R. Greenwood

Date :

5/18/00

Concur :

C. Soderqvist

Date :

5-19-00

Procedure: PNL-ALO-226

Measured Concentrations (ug/ml) with 1-sigma error

ALO ID Client ID	NH ₃ Error %
00-1701	1.83
AP-101 Comp	9%
00-1701Dup	1.70
AP-101 Comp	10%
00-1701 T	1.61
AP-101 Comp	10%
RSD	6%
MDL	0.2

Date May 19, 2000

To D. Kurath

From

M. Urie Subject Cyanide Results for AP-101 Sample

RPL Number	Sample ID	CN ($\mu\text{g} / \text{ml}$)	RPD (%)	RSD (%)	Spike Rec (%)
00-1701	AP-101 Sample	5.81	---	---	---
00-1701 D	AP-101 Duplicate	5.56	4.4	---	---
00-1701 T	AP-101 Triplicate	5.73	---	2.2	---
00-1701 MS	AP-101 Matrix Spike	---	---	---	95

The CN results for AP-101 composite samples analyzed on May 11, 2000 per ASR 5778 are reported in the Table above. The composite samples were transferred from the Shielded Analytical Laboratory and prepared for distillation and analyzed at the CN Workstation. The AP-101 samples were distilled with the addition of sulfamic acid to ensure there would be no interference from high nitrates present in the sample. The samples were analyzed using a Lachat QuickChem AE Autoanalyzer (WC36517). The reporting limits are estimated to be approximately 0.25 μg CN/ml based on the sample quantity distilled. No Quality Control or other measurement problems were encountered.

An independent mid-range calibration check solution run at the beginning, middle, and end of the analysis batch gave an average recovery of 100%. These calibration check standards ranged from 99% recovery to 100% recovery which is within the 85% to 115% acceptance criteria of the governing QA plan.

The AP-101 composite was analyzed in triplicate. The Relative Percent Difference (RPD) between the Sample and Duplicate was 4.4% and the Relative Standard Deviation (RSD) for the triplicates was 2.2%. Based on the RPD and RSD, the measurement precision meets the Quality Control criteria established by Table 4 of the ASR.

For the liquid AP-101 composite, the Laboratory Control Sample (LCS) was a distilled Blank Spike sample. The liquid LCS recovery was 101%, well within the Quality Control parameters established by Table 4 of the ASR. Beside the liquid LCS, as solid LCS was also processed. The solid LCS was analyzed at 112 $\mu\text{g/g}$ and 165 $\mu\text{g/g}$, well within the certified advisory range of 77 $\mu\text{g/g}$ to 301 $\mu\text{g/g}$. However, the solid LCS does not meet the ASR Quality Control criteria.

The spike recovery for the spiked AP-101 composite was 95%, well within the acceptance criteria of 75% to 125%.

All sample preparation sheets, standard preparation information, and analytical data are included with this report.

Distribution

No. of
Copies

No. of
Copies

OFFSITE

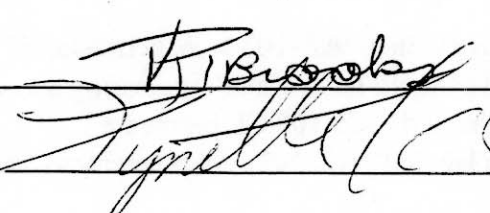
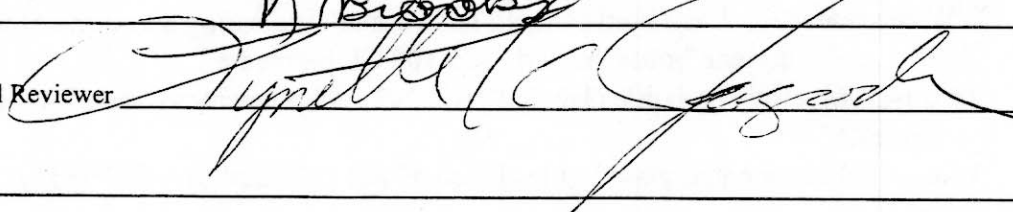
ONSITE

2 DOE/Office of Scientific and Technical
Information

5 British Nuclear Fuels, Limited
M. E. Johnson (4) BN-FL
A. Thompson BN-FL

19 Pacific Northwest National Laboratory
P. R. Bredt P7-25
J. A. Campbell P8-08
O. T. Farmer P8-08
S. K. Fiskum P7-22
L. R. Greenwood P7-22
D. E. Kurath P7-28
G. J. Lumetta P7-22
G. M. Mong P8-08
E. V. Morrey P7-28
R. T. Ratner P7-22
C. Z. Soderquist P7-22
R. G. Swoboda P7-22
M. W. Urie P7-22
J. J. Wagner P7-22
Technical Report Files (5) K1-06

Appendix A: CUF Filtration, Washing and Leaching Test Instructions and Laboratory Record Book

PNNL Test Instruction		Document No.: BNFL-TP-29953-076 Rev. No.: 0
Title: HLW Filtration and Caustic Leaching of AZ-102		
Work Location: RPL SFO HLRF	Page 1 of 19	
Author: Kenneth G. Rappé	Effective Date: Upon Final Approval Supersedes Date: New	
Use Category Identification: Information		
Identified Hazards: <input type="checkbox"/> Radiological <input type="checkbox"/> Hazardous Materials <input type="checkbox"/> Physical Hazards <input type="checkbox"/> Hazardous Environment <input type="checkbox"/> Other:	Required Reviewers: <input checked="" type="checkbox"/> Technical Reviewer <input type="checkbox"/> SFO Manager	
Are One-Time Modifications Allowed to this Test Instruction? <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <small>NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.</small>		
On-The Job Training Required? <input type="checkbox"/> Yes or <input checked="" type="checkbox"/> No FOR REVISIONS: Is retraining to this procedure required? <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No Does the OJT package associated with this procedure require revision to reflect procedure changes? <input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A		
Approval	Signature	Date
Author		1/8/00
Technical Reviewer		Jan 8, 2000

1.0 Applicability

This test instruction is to be used to perform the testing of the Cell Unit Filter (CUF) in the HLRF A-cell with 381 grams of AZ-102 slurry, which contains approximately 127.6 grams of undissolved solids.

2.0 Supporting Documents

This test instruction is not a stand-alone document. It will be used in conjunction with PNNL Operating Procedure BNFL-TP-29953-020, which contains the necessary procedural information for the safe operation of the CUF. It is also linked to PNNL Test Plan No. BNFL-TP-29953-069, which contains an overall description of the project, ES&H compliance, emergency response, and the hazards assessment and mitigation.

3.0 Responsible Staff

The staff responsible for executing this test plan are:

- Task Manager – Kriston Brooks
- SFO Manager – Randy Thornhill
- Test Engineers – Kriston Brooks, Ken Rappé, Lynette Jagoda
- Hot Cell Technician – Mac Zumhoff, Ralph Lettau, Don Rinehart
- Radiological Control Technician

4.0 Materials, Equipment, Supplies and Reagents Needed

4.1 Materials Required

- AZ-102 Samples “A,” “MU,” and “GL.” AZ-102 Recovery Jar.
- One 250 mL glass jar labelled “AZ-102 Initial Feed”
- Twenty-seven 20-mL glass scintillation vials for filtrate and slurry samples, pre-labeled as follows:
 - CUF-AZ102-001 through CUF-AZ102-027.
- Two 1-L bottles, labeled “AZ-102 Filtrate#1” and “AZ-102 Filtrate#2” with mark(s) indicating 970 milliliters.
- Two 1-L polyethylene bottle labeled “CUF AZ-102 First Wash.” It should be marked with a line for 1-L volume.
- Three 1-L polyethylene bottles labeled “AZ-102 WW permeate#1” through “AZ-102 WW permeate#3”. Each bottle should have a mark indicating a volume of 900-mL.
- One 1-L polyethylene bottle labeled “AZ-102 CL permeate.”
- Two 1-L polyethylene bottles labeled “AZ-102 CW permeate#1” and “AZ-102 CW permeate#2.”
- Two 1-L plastic-coated glass bottles labeled “AZ-102 final sample” and “AZ-102 decant.”
- Three graduated centrifuge tubes.
- Tubing for de-watering mode.
- Two 10 liter containers, one labeled for the alkaline rinses and the other labeled for the acidic rinses.
- Containers for draining from the bottom of the pump and from the sample valve.
- One 2-L bottle labeled “tank overflow container.”

- Tubing to go from valve V-10 to the caustic leaching/washing vessel.
- 10 liters of 0.2-micron filtered DI water for washing the CUF upon completion of AZ-102 processing.

4.2 Equipment

- 4000 gram balance
- Video tape
- pH paper
- Hand held camera. Used for making difficult readings and volume estimations.
- Stopwatch
- Calculator
- CUF Ultrafiltration system with 100 mL plug in place
- 1000 watt Chiller

4.3 Reagents Needed

All containers that will be put into the cell should be labeled with "AZ-102" and "CUF testing."

- 1-L of filtered, distilled water labeled "start-up water."
- 1.62 liters of 0.01-M NaOH_{aq} labeled "5 wt % dilution 1."
- TBD mL (837 g - ~~rinse bottle liquid~~ ^{recovery jar liquid} g) of 0.01-M NaOH_{aq} labeled "5 wt % dilution 2."
- 2.7 liters of 0.01-M NaOH_{aq} labeled "water wash solution" split up into three separate 1-L containers containing 900-mL of solution in each.
- NaOH_{aq} labeled "caustic leach solution." Amount and molarity to be determined. Will be approximately 600 mL.
- 100 mL bottle of 3 M NaOH_{aq} labeled "3M caustic rinse." Determine mass of caustic in bottle.
- Two containers of 0.01-M NaOH_{aq} labeled "caustic wash solution#1" and "caustic wash solution#2." Amount to be determined. Will be approximately 1600 mL of solution in *each* container.
- 1 liter of filtered, distilled water labeled "AZ-102 CUF rinse."
- 1 liter of 1-M HNO_{3aq} labeled "AZ-102 CUF wash acid."
- 1 liter of filtered, distilled water labeled "shut-down water."

4.4 Other Supplies

1. Workplace Copy of Operating Procedure BNFL-TP-29953-020
2. Extra Copies of Data Sheets 1, 2, and 3
3. Laboratory Record Book
4. DAS disk for recording data

5.0 Test Instructions

The laboratory record book (LRB) shall be used to record other testing information as required by this procedure and all test conditions not stated by this procedure.

Cross-contamination between samples and contamination of samples from outside sources must be minimized at each step. Use new tools and bottles for each sample as much as possible. Those tools that are reused should be washed and rinsed prior to reuse.

Keep all test materials in sealed containers as much as possible to prevent them from drying. Due to the shortage of test sample, *do not* remove any additional slurry other than what is specifically requested for in this test instruction.

5.1 Pre-start

5.1.1 Inventory materials, equipment, supplies, and reagents to ensure all required items are available. Assure that all materials have been modified for remote handling.

5.1.2 Do the following and initial and date when each item is completed.

✓ Review PNNL Operating Procedure BNFL-TP-29953-020.

✓ Review the work instructions in this document.

5.1.3 Weight AZ-102 Sample A and tare weigh the bottle labeled "AZ-102 Initial Feed"

AZ-102 Sample A

Total 788.56 g
w/ cap & w/o metal clamp

Tare AZ-102 Initial Feed

Total 352.33 g (w/ cap)
327.89 g (w/o cap)

5.1.4 Transfer 284 g of AZ-102 from AZ-102 Sample A to AZ-102 Initial Feed.

Reweigh both containers. Any solids remaining on the tools used for the transfer should be saved and will be rinsed into the CUF.

Not performed. See LRB.

AZ-102 Sample A

Total 466.57 g

AZ-102 Initial Feed

Total 328.54 g

5.1.5 Conduct the "0.0 Pre-Start" operations in BNFL-TP-29953-020. Replace the overflow container with the bottle labeled "tank overflow container."

** Steps 5.1.6 through 5.1.12 have already been completed. They will remain in the instruction for reference purposes only. Operations should begin with step 5.1.13.

5.1.6 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with lay-up solution currently in CUF with one variation: V4 the filtrate control valve should be closed. Run CUF for 5 minutes between 4 and 6 gallons per minute (gpm).

5.1.7 Conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.1.8 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water.

5.1.9 Perform "6.0 Back-pulsing" operations in BNFL-TP-29953-020.

5.1.10 Shut off the system and conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.1.11 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 using the liquid in the bottle labeled "start-up water".

5.1.12 Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions below. Filtrate flow rate should be monitored and data collected in the operating procedure. Each test should be performed for only 20 minutes and the system should be back-pulsed. After each condition, the test engineer should initial and date the table below.

Condition	Flowrate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	4.20	30	
2	4.20	55	
3	4.20	70	

5.1.13 Shut off the system and conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.2 Start-Up

5.2.1 Obtain the following information:

M&TE List:

✓ Balance 1: (Small)

Calib ID 326-06-01-061

Calib Exp Date 8/2000

Location A-cell South

✓ Balance 2: (large)

Calib ID 326-06-01-054

Calib Exp Date 8/2000

Location A-cell north

TC 1 Calib ID/Date 3003/8/00 P-201 Calib ID/Date 325-30-2 / Exp. 1/2000

P-202 Calib ID/Date 325-30-3 Exp. 1/2000 P-203 Calib ID/Date 3054 / Jan. 2001 Exp.

5.2.2 Weigh the jars containing the AZ-102 samples and the "5 wt% dilution 1." Record their weights below.

AZ-102 Sample MU

Total 175.320 g

AZ-102 Sample GL

Total 180,833 g

"5 wt% dilution 1"

Total See LRB g

5.2.3 Conduct the "1.0 Start-Up" operations in BNFL-TP-29953-020 starting with approximately one-half of the material in "5wt% dilution 1." Once that is pumping, add the contents of AZ-102 Initial Feed. Shake the jar containing the AZ-102 sample thoroughly before adding it to the slurry reservoir. Use the remaining "5wt% dilution" material to recover any solids left in the sample jar that cannot be transferred by shaking. Pay close attention to the level of material in the CUF.

5.2.4 Record the weights of the empty jars and lids in the spaces provided below. Calculate the amount of material remaining in the sample jar. If the difference is greater than ~ 2 g or significant solids are visible, further solids recovery is warranted. Record the method of recovery in the LRB.

AZ-102 Sample A

AZ-102 Initial Feed

"5wt% dilution"

466.57 g
788.56 g
321.99 g

Jar + ~~Lid~~ 328.54 g
Tare 327.89 g
Total 0.65 g

Jar + Lid See LRB g
Tare See LRB g
Total 1849.86 g

5.2.5 Record the level in the slurry reservoir.

Height _____ inches 22.2 cm 6" = 15 cm

5.3 Operation

First Test Matrix

5.3.1 Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions below. Filtrate flow rate should be monitored and data collected as specified in the operating procedure. After each condition, the test engineer should initial and date the table below.

Condition	Flow rate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	4.20	50	<u>LRB</u> <u>7/10/00</u>

2	3.13	30	FR 1/10/00
3	5.23	70	KGR 1/10/00
4	5.23	30	KGR 1/10/00
5	3.13	70	-
6	4.20	50	KGR 1/10/00
7	Optimum from 1 - 6	Optimum from 1 - 6	19/6/00

~ 4.5 (best each row)

30

- KGR 5.3.2 Using the rheometer sample cup, obtain 40 mL of slurry material for rheological measurements following "7.0 Slurry Sampling" in BNFL-TP-29953-020. This may require as many as eight samplings to obtain this material. Perform rheological measurement per Table 4 in BNFL-TP-29953-069.

Viscometer

Make HAAKE

Model M5 RVRC 20

Geometry mv1

24.7°C

- 5.3.3 Tare-weigh the 1-L bottle labeled "AZ-102 Filtrate#1."

18:29 pm Weight of empty "AZ-102 Filtrate#1" with lid 102.626 g
1A

- 5.3.4 Pre-weigh two of the labeled 20-mL glass scintillation vials and record its weights. Mid-way through the following step (5.3.5), obtain two filtrate samples of at least 20 grams following "8.0 Filtrate Sampling" in BNFL-TP-29953-020. If required, more than two sample vials may be used. Record the weights and sample numbers in Data Sheet 3. These will be used for chemical and radiochemical analyses.

- 18:45 - 2 back pulses -
5.3.5 Conduct the "4.0 Operation during Ultrafilter De-watering Mode" operations in BNFL-TP-29953-020 using the optimum conditions from step 5.3.1. Fill "AZ-102 Filtrate#1" to the 970 mL mark, or as high as possible if this mark is unattainable.

- 5.3.6 Replace the lid on the bottle and weigh.

Weight of "AZ-102 Filtrate#1" with lid 1060.60 g

Total weight of material removed in 957.97 g

AZ-102 Filtrate#1 + 2 samples

- 5.3.7 Record the level in the slurry reservoir.

Height ~1-2" inches

19/6 video

- 5.3.8 Weigh the bottle labeled "5 wt% dilution 2" and the AZ-102 recovery jar. Calculate the weight of liquid/slurry in the recovery jar.

Tare Weight of "5 wt% dilution 2" with lid NA g

Weight of AZ-102 recovery jar 735.69 g

Tare Weight of AZ-102 recovery jar 351.87 g

Weight of liquid/slurry in recovery jar 383.82 g

visually settle
~ 1050 Solids
~ 9050 Supernate
see vial

5.3.9 Calculate and add the proper weight of 0.01M NaOH to the bottle labeled "5 wt% dilution 2."

Weight required to be added to CUF 837 g

Weight of liquid/slurry in recovery jar 383.82 g

Weight needed in "5 wt% dilution 2" 453.18 g

Actual weight of "5 wt% dilution 2" added 453.19 g

307.31
145.88

Pre - MU = 175.3205
Pre - GL = 180.835
5.3.10 Add the remaining slurry material from "AZ-102 Sample A", "AZ-102 Sample MU," and "AZ-102 Sample GL" to the CUF. *Rinse Composite*

5.3.11 Use the AZ-102 recovery jar and 5 wt% dilution 2 liquids to rinse out the three AZ-102 sample jars. Pour this material into the CUF. *finished @ ~ 2030*

5.3.12 Weigh the empty containers.

When looked in CUF no slurry movement - Plugs - had to pulse manually to break - allowing 130min to mix & break-up any flocs/aggr. *OK*

Weight of "AZ-102 Sample A" 465.10 g

Weight of "AZ-102 Sample MU" 134.485 g

Weight of "AZ-102 Sample GL" 134.169 g

Weight of recovery jar 351.87 g

Weight added to CUF

Weight of "AZ-102 Sample A" 465.10 g

Weight of "AZ-102 Sample MU" 134.485 g

Weight of "AZ-102 Sample GL" 134.169 g

Weight of recovery jar 735.84 g

5.3.13 Tare-weigh the 1-L bottle labeled "AZ-102 Filtrate#2."

Weight of empty "AZ-102 Filtrate#2" with lid 100.361 g

5.3.14 Pre-weigh two of the labeled 20-mL glass scintillation vials and record its weights. Mid-way through the following step (5.3.15), obtain two filtrate samples of at least 20 grams following "8.0 Filtrate Sampling" in BNFL-TP-29953-020. If required, more than two sample vials may be used. Record the weights and sample numbers in Data Sheet 3. These will be used for chemical and radiochemical analyses.

Height before de-watering 4-4.5"
(close to 4)

* Having lots of plugging problems.

5.3.15 Conduct the "4.0 Operation during Ultrafilter De-watering Mode" operations in BNFL-TP-29953-020 using the optimum conditions from step 5.3.1. Fill "AZ-102 Filtrate#2" to the 970 mL mark, or as high as possible if this mark is unattainable. *R 1/10/00*

5.3.16 Replace the lid on the bottle and weigh.

Weight of "AZ-102 Filtrate#2" with lid *828.18* g
Total weight of material removed *727.819* g

5.3.17 Record the level in the slurry reservoir.

Height *~1"* inches

very thick - not mixing well in tank
added back ~200 ml of permeate from filtrate #2
New Height ~2-2.5"

Second Test Matrix

5.3.18 Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions below. Filtrate flow rate should be monitored and data collected as specified in the operating procedure. After each condition, the test engineer should initial and date the table below, and check the level in the slurry reservoir (video-taping periodically, as necessary).

Condition	Flowrate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	<i>4.20 2.6</i>	50	<i>JTB 1/11/00</i>
2	<i>3.13 2.2</i>	30	<i>JTB 1/11/00</i>
3	<i>5.23 2.0</i>	70	<i>JTB 1/11/00</i>
4	5.23	30	<i>JTB 1/11/00</i>
5	3.13	70	<i>Not performed</i>
6	4.20	50	<i>JTB 1/11/00</i>

5.3.19 Obtain two slurry samples of 20 and 10 grams each, respectively, following "7.0 Slurry Sampling" in BNFL-TP-29953-020 and using the pre-labeled sample vials.

These will be used for physical property and particle size distribution analyses. *~ 11:45 pm*
Record the weights and sample numbers in Data Sheet 3.

5.3.20 Using the rheometer sample cup, obtain 40 mL of slurry material for rheological measurements following "7.0 Slurry Sampling" in BNFL-TP-29953-020. This may require as many as eight samplings to obtain this material. See Table 4 in BNFL-TP-29953-069 for a description of this process. *JTB 1/10/00*

5.3.21 Adjust the flow to < 2 gpm and the pressure to < 10 psig ~~or turn off the pump~~ while the rheology of the material is measured. Pour the 40-mL used for rheological measurements back into the CUF. Perform rheological measurement per Table 4 in BNFL-TP-29953-069. K7B 4/19/00

5.3.22 Increase the flow and pressure to its optimal value and repeat step 5.3.20 and 5.3.21 for a second 40-mL slurry sample.

Three Water Washes

5.3.23 Weigh all three one-liter bottles labeled "water wash solution."

Bottle with solution (Solution only)	#1 <u>896.53</u> g	#2 <u>905.42</u> g	#3 <u>895.80</u> g
--	-----------------------	-----------------------	-----------------------

5.3.24 Obtain the tare weight of the three empty two-liter bottles labeled "AZ-102 WW permeate#1" through "AZ-102 WW permeate#3."

	permeate#1	permeate#2	permeate#3
Empty bottle	<u>100.00</u> g	<u>100.29</u> g	<u>100.58</u> g

5.3.25 Add the first bottle of "water wash solution" to the slurry reservoir.

5.3.26 Conduct the "4.0 Operation during Ultrafilter De-watering Mode" operations in BNFL-TP-29953-020 using the optimum conditions from step 5.3.12, acquiring flux measurements periodically. Fill "AZ-102 WW permeate#1" to the 900-mL mark. Add the second bottle of "water wash solution", and begin filling "AZ-102 WW permeate#2." Add the third bottle of "water wash solution" once the 900-mL mark is reached in the second permeate bottle. Continue dewatering into "AZ-102 water wash permeate#3," filling it to its 900-mL mark, or as high as possible if the 900-mL mark is unattainable. Following each dewatering (after each 900-mL mark is reached), check the slurry height. Video tape if necessary. 18 K7B 4/19/00

5.3.27 Weigh the three empty one-liter bottles labeled "water wash solution."

Bottle without solution	#1 <u>N/A</u> g	#2 <u>N/A</u> g	#3 <u>N/A</u> g
Total added	<u>896.53</u> g	<u>905.42</u> g	<u>895.80</u> g

5.3.28 Weight the three one-liter bottles labeled "AZ-102 WW permeate#1" through "AZ-102 WW permeate#3."

	permeate#1	permeate#2	permeate#3
with permeate	<u>978.57 g</u>	<u>981.14 g</u>	<u>1054.65 g</u>
Total permeate	<u>978.57 g</u>	<u>980.85 g</u>	<u>954.07 g</u>

5.3.29 Obtain the tare weight of a graduated centrifuge tube.

Tare weight of centrifuge tube = 6.4899 g

5.3.30 Obtain 1 slurry sample of at least 10 grams following "7.0 Slurry Sampling" in BNFL-TP-29953-020 and using the graduated centrifuge tube. Determine the weight of the sample ($W_{t_{sample}}$) and the total sample volume (V_{sample}). Centrifuge for 5 minutes, remove, and determine volume of centrifuged solids (V_{solids}).

Weight (tube and sample) = 15.91 g

$W_{t_{sample}} =$

5:40 pm Weight (tube and sample) - weight of centrifuge tube = 9.4201 g
 wt of empty tube after return of sample to CUF 6.817 g

$V_{sample} =$ 8.6 mL

$V_{solids} =$ 1.6 mL

$V_{slurry/tank} =$ 0.186 mL

slurry wt = 0.321 g

2.1
 F.B. 1/11/00

5.3.31 Calculate the volume of required leaching solution, ~~3M~~ NaOH_{aq} and place in "caustic leach solution." Place a mark indicating this volume on the bottle labeled "AZ-102 CL permeate#1". V_{slurry} represents the volume of the slurry.

Volume of ~~3M~~ NaOH_{aq} to be used =

$3 * V_{slurry/tank} * (V_{solids} / V_{sample}) =$ 483 mL

see LRB pg 98

5.3.32 Record the level in the slurry reservoir.

Height ~1" / 1.5 inches

5.3.33 Obtain one slurry sample of at least 20 grams following "7.0 Slurry Sampling" in BNFL-TP-29953-020 and using the pre-labeled sample vials. This will be used for physical analyses. Record the weight and sample number in Data Sheet 3.

6:25 pm
 1/11/00

Caustic Leach

5.3.34 Measure the Tare of the empty caustic leach container.

-No lid / no baffles etc.
2.1

Tare container 1124.41 g

5.3.35 Drain the system of slurry following "10.0 Draining the System" with the following changes: rather than placing a container underneath sample port V10 to

collect slurry, tubing will be connected to the sample port and flow directed into the caustic leaching/washing container. - Rinsed sample port into caustic leach container approx 10ml added (DI H₂O)

- 5.3.36 Prepare the NaOH_{aq} solution. The volume of this solution was determined above. The concentration is to be sufficient to create a 3M NaOH solution above the centrifuged solids layer.

Volume NaOH solution required = 483 ml g
 Molarity of NaOH solution = 7.37 M
 Bottle with 3-M NaOH_{aq} = 676.49 g - Empty 67.331 g
 Both solutions both tare 66.93 g

- 5.3.37 Add the above volume of solution to the slurry reservoir and pump through the system for five minutes. Drain as described above (5.3.35) into the caustic-leaching vessel. Determine the weight of 3-M NaOH_{aq} added.

Leach 2800.66 g
 Container with 3-M NaOH_{aq} = 67.331 g
 Level in leaching container 2" = ~1600ml slurry
 2114 pm start 5 min
 21 609.159 g added
 0.4018 lb in bottle
 2154

- 5.3.38 Begin heating the slurry and set the controller temperature to 85°C. Secure the top on the leaching/washing vessel along with the attached mixer. Initiate steady mixing. Wait until the slurry temperature reaches 85°C and note the time. Leaching will continue for 8 hours from this time.

Time 85°C is reached 10:15 AM/PM 1/11/00
 Estimated Shutdown time (above time plus 8 hours) 6:05 AM/PM 1/12/00
 Shut off CUF - 6:33 AM 1/12/00

- 5.3.39 Obtain the tare weight of the empty one-liter bottle labeled "AZ-102 CL permeate."

AZ-102 CL permeate
 Empty bottle 67.000 g

Rinsed Container w/

- 5.3.40 Begin heating the CUF at the end of the caustic leach. When leaching is complete, stop the heating/mixing the caustic leach. Measure weight and level of full container.

Container w/slurry 2721.54 g
 Level 1 32
32

- 5.3.41 Add the slurry to the slurry reservoir. Pump until the temperature reaches 85±5°C.

- 5.3.42 Calculate water lost during leaching.

Full container pre-leach 2800.66 g

Full container post-leach	<u>272.54</u>	g
Amount of water lost	<u>79.12</u>	g
	+ 28.539 already added	

5.3.43 Add amount of water equal to that lost into the container, swirl and transfer into the slurry reservoir.

Actual Water Added to CUF 81.46 g

Note if too many solids remain in the container, rinse the caustic leach vessel with an additional 100-mL of 3-M NaOH_{aq} to remove any residual solids on the bottom or sides and add to the reservoir. Determine the weight of 3-M NaOH_{aq} added.

Container with 3-M NaOH _{aq} =	<u>Not performed</u>	g
Container without 3-M NaOH _{aq} =	<u>Not performed</u>	g

5.3.44 Pre-weigh two of the labeled 20-mL glass scintillation vials and record their weights. Mid-way through the following step (5.3.45), obtain two filtrate samples.

5.3.45 Conduct the "4.0 Operation during Ultrafilter De-watering Mode" operations in BNFL-TP-29953-020 using the optimum conditions from step 5.3.18. Obtain two filtrate samples of at least 20 grams following "8.0 Filtrate Sampling" in BNFL-TP-29953-020. If required, more than two sample vials may be used. Record the weights and sample numbers in Data Sheet 3. These will be used for chemical and radiochemical analyses. Fill "AZ-102 CL permeate" to the mark made in step 5.3.31, or as high as possible if mark is unattainable.

5.3.46 Weight the full one-liter bottle labeled "AZ-102 ^{Caustic Add} ~~CL permeate~~."

	AZ-102 CL permeate
With permeate	<u>596.58</u> g

5.3.47 Record the level in the slurry reservoir.

Height not done inches

5.3.48 Obtain three slurry samples of at least 20 grams each following "7.0 Slurry Sampling" in BNFL-TP-29953-020 and using the pre-labeled sample vials. These will be used for chemical and physical analyses. Record the weights and sample numbers in Data Sheet 3.

First Caustic Wash

5.3.49 Drain the system of slurry following "10.0 Draining the System" with the following changes: rather than placing a container underneath sample port V10 to collect slurry, tubing will be connected to the sample port and flow directed into the caustic leaching/washing container.

Level with slurry 13 1/16 - inch
 Volume of slurry 702.3 mL

- 5.3.50 Add sufficient volume of 0.01-M NaOH_{aq} to the slurry reservoir from "caustic wash solution#1" to make 2400 mL total volume (slurry and pump through the system for five minutes. Drain as described above (5.3.34) into the caustic-leaching vessel. Determine the weight of 0.01-M NaOH_{aq} added. Determine the weight of full leaching container. 1/10/00

labeled "AZ-102 CW permeate #1" ←
 Volume 0.01-M NaOH_{aq} required (2400-slurry volume) 1400 mL
 Volume 0.01-M NaOH_{aq} added 1400 mL
 Weight of Full Container with 0.01-M NaOH_{aq} 1659.86 g tare
 Level in leaching container 2 - 7/8 - inches 2.288 - liters
 Mass in leaching container 3520.60 g

- 5.3.51 Begin heating the slurry and set the controller temperature to 85°C. Secure the top on the leaching/washing vessel along with the attached mixer. Initiate steady mixing. Wait until the slurry temperature reaches 85°C and note the time. Leaching will continue for 8 hours from this time.

Time 85°C is reached 1 2:54 AM/PM
 Shutdown time (above time plus 8 hours) 08:54 AM/PM

- 5.3.52 Obtain the tare weight of the empty one-liter bottle labeled "AZ-102 CW permeate#1."

permeate#1
 Empty bottle 267.63 g

- 5.3.53 Begin heating the CUF at the end of the caustic wash. When washing is complete, stop the heating/mixing the caustic wash. Measure weight and level of full container. 2100

Container w/slurry 3415.80 g
 Level 2 - 7/8 "

- 5.3.54 Add the slurry to the slurry reservoir. Pump until the temperature reaches 80°C.

- 5.3.55 Calculate water lost during leaching.

Full container pre-leach 3520.60 g
 Full container post-leach 3415.80 g
 Amount of water lost 104.80 g

- 5.3.56 Add an amount of water equal to that lost into the container, swirl, and transfer into the slurry reservoir.

Actual water added to CUF 105.25 g

Note if too many solids remain in the container, rinse with an addition 100 mL of 0.01 M NaOH solution. Pour into the CUF. Determine the amount added.

Additional Water added to the CUF Not performed g

- ✓ 5.3.57 Pre-weigh two of the labeled 20-mL glass scintillation vials and record their weights. Mid-way through the following step, obtain two filtrate samples.
- ✓ 5.3.58 Conduct the "4.0 Operation during Ultrafilter De-watering Mode" operations in BNFL-TP-29953-020 using the optimum conditions from step 5.3.18, recording flux measurements periodically. Obtain two filtrate samples of at least 20 grams following "8.0 Filtrate Sampling" in BNFL-TP-29953-020. If required, more than two sample vials may be used. Record the weights and sample numbers in Data Sheet 3. These will be used for chemical and radiochemical analyses. Fill "AZ-102 CW permeate#1" to the amount added in step 5.3.48, or as high as possible if mark in unattainable.

50 1/10/00

5.3.59 Record the level in the slurry reservoir.

Height Not Performed inches (video taped)

Second Caustic Wash

- 5.3.60 Drain the system of slurry following "10.0 Draining the System" with the following changes: rather than placing a container underneath sample port V10 to collect slurry, tubing will be connected to the sample port and flow directed into the caustic leaching/washing container.

Tare container 1124.41 g

Level with slurry Not performed - taken from weight & volume below

Volume of slurry Not performed - taken from weight & volume below

- 5.3.61 Add sufficient volume of 0.01-M NaOH_{aq} to the slurry reservoir from "caustic wash solution#2" to make 2400 mL total volume (slurry and and pump through the system for five minutes. Drain as described above (5.3.34) into the caustic-leaching vessel. Determine the weight of 0.01-M NaOH_{aq} added. Determine the weight of the full leach container.

35 1/10/00

Volume 0.01-M NaOH_{aq} required (2400-slurry volume) 1400 mL

Volume 0.01-M NaOH_{aq} added 1400 mL

Weight of Full Container with 0.01-M NaOH_{aq} 1661.98 g

Level in leaching container 2 11/16" (2144 mL)

Weight of Full Leach Container 3259.25

- 5.3.62 Begin heating the slurry and set the controller temperature to 85°C. Secure the top on the leaching/washing vessel along with the attached mixer. Initiate steady mixing. Wait until the slurry temperature reaches 85°C and note the time. Leaching will continue for 8 hours from this time.

Time 85°C is reached 4:13 AM → 12:13
 Shutdown time (above time plus 8 hours) 12:13 AM

- 5.3.63 Obtain the tare weight of the empty one-liter bottle labeled "AZ-102 CW permeate#2."

permeate#2
 Empty bottle 261.982 g

- 5.3.64 Begin heating the CUF at the end of the caustic wash. When washing is complete, stop the heating/mixing the caustic leach.

Container w/slurry 3237.10 - 100.068g
 Level not taken

- 5.3.65 Add the slurry to the slurry reservoir. Pump until the temperature reaches 80°C.

- 5.3.66 Calculate the water lost during heating.

Full container pre-leach	<u>3259.25</u>	g
Full container post-leach	<u>3137.032</u>	g
Amount of water lost	<u>122.218</u>	g

- 5.3.67 Add an amount of water equal to that lost into the container, swirl, and transfer into the slurry reservoir.

Actual water added to CUF 100.068 g

Note if too many solids remain in the container, rinse with an addition 100 mL of 0.01 M NaOH solution. Pour into the CUF. Determine the amount added.

Additional Water added to the CUF 150.092 g

- 5.3.68 Pre-weigh two of the labeled 20-mL glass scintillation vials and record their weights. Mid-way through the following step, obtain two filtrate samples.

Measure filtrate flux initial, middle, end (over course of 10 minutes).

- 5.3.69 Conduct the "4.0 Operation during Ultrafilter De-watering Mode" operations in BNFL-TP-29953-020 using the optimum conditions from step 5.3.18, recording flux measurements periodically. Obtain two filtrate samples of at least 20 grams following "8.0 Filtrate Sampling" in BNFL-TP-29953-020. If required, more than

two sample vials may be used. Record the weights and sample numbers in Data Sheet 3. These will be used for chemical and radiochemical analyses. Fill "AZ-102 CW permeate#2" to the amount added in step 5.3.61, or as high as possible if mark in unattainable.

5.3.70 Record the level in the slurry reservoir.

Height not done inches

two #18 9/13/00
~~one~~ 2

5.3.71 Weight the ~~two~~ ^{one} full one-liter bottles labeled "AZ-102 CW permeate#1" and "AZ-102 CW permeate#2."

stet. #18 1/13/00

	permeate#1	permeate#2
With permeate	<u>1759.69 g</u>	<u>1801.32 g</u>
Tare	<u>267.63 g</u>	<u>261.982 g</u>
	<u>1492.06</u>	<u>1539.338</u>

Testing Finale

(Call Paul - if he is available do this. If not, collect a 80 g sample for him.)

*Acquiring
80ml of sample
for testing
later.*

5.3.72 Using the rheometer sample cup, obtain 40 mL of slurry material for rheological measurements following "7.0 Slurry Sampling" in BNFL-TP-29953-020. This may require as many as eight samplings to obtain this material.

5.3.73 Adjust the flow to < 2 gpm and the pressure to < 10 psig or turn off the pump while the rheology of the material is measured. The 40-mL of material will be recovered at a later time. Determine weight of material used.

5.3.74 Increase the flow and pressure to its optimal value and repeat step 5.3.62 and 5.3.63 for a second 40-mL slurry sample.

72 #18 1/19/00

73 1/10/00

5.3.75 Obtain four slurry samples of at least 10 (1) & 20 (3) grams each, respectively, following "7.0 Slurry Sampling" in BNFL-TP-29953-020 and using the pre-labeled sample vials. These will be used for chemical and physical analyses, and particle size distribution. Record the weights and sample numbers in Data Sheet 3.

*OK
6:33 PM*

Draining and Rinsing the System

1L glass (w/ plastic coating)

5.4.1 Tare-weigh the 2-L bottle labeled "AZ-102 final sample."

Weight of empty "AZ-102 final sample" with lid 520.37 g

5.4.2 Drain the system of slurry following "10.0 Draining the System" into "AZ-102 final sample."

Weight of full "AZ-102 final sample" with lid 1097.35 g

*576.98 g
in Final
Sample*

Tare AZ-102 17 washed solids 354.756 g

Full AZ-102 washed solids 673.04 g

*318.284 g
removed*

- 5.4.3 Add the 1-L of filtered, distilled water in "AZ-102 CUF rinse" to the reservoir. Open V4 and pump through the entire system for 5 minutes.

Weight of full "AZ-102 CUF rinse" _____ g
 Weight of empty "AZ-102 CUF rinse" _____ g
 Weight of "AZ-102 CUF rinse" _____ g

See LRB
for details

- 5.4.4 Back-pulse the system once following "6.0 Back-Pulsing."

- 5.4.5 Shut off pump and drain the system following "10.0 Draining the System."

Weight of full "CUF rinse" See LRB _____ g

- 5.4.6 Conduct the "9.0 Rinsing the system" operation in BNFL-TP-29953-020 for the second and third rinses. The second rinse should be done with 2-L of filtered, distilled water, and the third rinse with 1-L of filtered, distilled water. The second and third rinses should be collected separately from the first in the alkaline rinse storage container. Filtrate flow rates should be checked with the third rinse to determine if an acidic rinse is required. The acidic solutions, if necessary, should be placed in a separate container.

- 6 Back pulse during 2nd rinse / None for 1st Rinse.

- 5.4.7 Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions below and the 1-L of filtered, distilled water in "shut-down water." Filtrate flow rate should be monitored and data collected in the operating procedure. Each test should be performed for only 20 minutes and the system should be back-pulsed. After each condition, the test engineer should initial and date the table below.

Condition	Flowrate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	4.20	30	KGR 1/14/00
2	4.20	35 40	KGR 1/14/00
3	4.20	70	

Opps Should have been 10, 20, 30 psig
1/14/00
7:45 pm

- 5.4.8 Conduct the "11.0 Shutting down" operation in BNFL-TP-29953-020.

- 5.4.9 Conduct the "12.0 Lay Up" operation in BNFL-TP-29953-020.

"AZ-102 Final Sample"

- 5.4.10 Allow the slurry to settle in the 2-L bottle. Analysis of the permeate from the second caustic wash, "AZ-102 caustic wash permeate#2," will determine if further washing is required. If no further washing is required, decant off the liquid on top of

the settled slurry into "AZ-102 decant" until 1 to 2 inches of liquid remain. Weigh the amount of liquid removed.

Weight of empty "AZ-102 decant" with lid _____ g
Weight of full "AZ-102 decant" with lid _____ g

5.4.11 Remove and weigh all appropriate samples from all permeate bottles.

6.0 Sample Analysis

The point of contact for physical property sample analysis of the slurry samples is Paul Bredt. The point of contact for the sample analysis of the filtrate, wash, and filtered solids samples is Mike Urie and Rick Steele.

6.1 Slurry Sample Physical Analysis

The slurry samples taken for physical testing (except for particle size distribution analysis samples, which will go to Lab 312) will remain in the HLRF. These samples will be used for obtaining the following information: bulk density, supernatant density, particle size distribution, volume percent settled and centrifuged solids, and suspended solids loading. Each of these analyses will be done in duplicate. The viscosity of these samples was performed previously during testing in Section 5.0.

6.2 Chemical and Radiochemical Analysis

The following samples should be transferred to the SAL hot cells for prep work and analysis.

Liquid Samples

- One sample from the first de-watering step.
- One sample from the second de-watering step.
- One sample taken from the bottle labeled "AZ-102 water wash permeate#1"
- One sample taken from the bottle labeled "AZ-102 water wash permeate#2"
- One sample taken from the bottle labeled "AZ-102 water wash permeate#3"
- One sample taken during the AZ-102 caustic leach
- One sample taken during the first AZ-102 caustic wash #1
- One sample taken during the second AZ-102 caustic wash #2
- One sample taken from the bottle labeled "AZ-102 decant"

Slurry Samples

- One sample after three water washes
- Two samples after the caustic leach
- Two samples after the final dilute caustic wash

The following will be performed on these samples: TOC/TIC and IC on the original sample and acid digestion followed by GEA, Sr-90 analysis, ICP-MS for Tc-99, total alpha and ICP-AES. See BNFL-TP-29953-069 for further details.

AZ-102 Test

1/10/00

The CUF has been rinsed w/ HNO_3 / 0.2M citric acid to finish cleaning it after the AN-107 run.

It has been rinsed to neutral pH and the clean water flux measured. A new pressure gauge on permeate side was needed & the leak on the chilled water is fixed. We are concerned about whether the water we insert in the tank outside the cell all ends up in the CUF.

Weight of water ^(0.01M NaOH) outside	=	243.51g
Tare Weight of Container inside	=	102.32g
Weight of full container inside	=	<u>323.84g</u>
Weight of 0.01M NaOH inside		<u>221.52g</u>
Delta		21g

Weight of water outside _(0.01M) = 688.07g (#1)

Weight of (0.01M) NaOH outside 711.95g (#2)

We attempted to transfer the solids to a different container. In the process, material was being lost, dried on and getting spread everywhere. Instead we will send all of the material both in AZ-102 A and the other new container into the CUF.

We may have lost 2-3g dried solid on the cloths where we set the spatulas. We may have lost an additional ~~2-3~~ 3-4g on the spatulas.

Flow Mx+B settings:

M = 1.9901

B = -1.9987

Project No. 29953 Date of Work 1/10/00
 Entered By RP Brooks Date 1/10/00
 Disclosed To and Understood By _____
 Signed 1. _____ Date _____

AZ-102

2:10 am

Attached new pressure gauge.
 Drained CUF
 Turned on DAS

Added 106.09 g of 0.01 M NaOH to
 the AZ-102 Initial feed container (maybe 10g went
 to AZ-102 Sample A) - (from existing squirt bottle)

Added 221.52 g of 0.01 M NaOH to AZ-102 sample A

Weight of 788.58 g
 Container + 0.01 M NaOH. \rightarrow 686.26 g

Poured AZ-102 Initial Feed into CUF (w/ 106.09 g water)

Weight of 815.20 g
 Container + 0.01 M NaOH \rightarrow 712.88 g

Poured into AZ-102 Sample A to clean bottle.

4:02 Repeated as needed. Cleaned up spatulas as well as could.

AZ-102 Sample A has a lid that says
 "AZ-102 Recovered Solids Rin"
 Side says "AZ-102"

4:35 Started CUF
 - initially no flow & pressure built up in the inlet pressure gauge
 but not the outlet. Lots of flow variation/pressure variation -
 Added
 173.11 g of 0.01 M NaOH additional to get it to 5 wt%

4:52 Opened V4 and began flux measurements.
 Needed air stepper to get to 50 psi & still can't get
 to 4.2 gpm.


Project No. 29953 Date of Work 1/10/00
 Entered By KP Brooks Date 1/10/00
 Disclosed To and Understood By _____
 Signed 1. _____ Date _____
 2. _____ Date _____

AZ-102

- 5:20 Continues to creep up in pressure over time. It also appears that the flow rate is increasing over time. The flux is decreasing over time.
- 6:06 Started condition #2
- 6:16 Adjusted velocity down. Since 4.2 gpm wasn't achieved, we need to run the matrix at lower axial velocities.
- 7:23 System plugs at condition #3 such that pressure zooms up to 100 psi & then you have to back way down to get it to equilibrate. All the way down to 30 psi. Readjust & the pressure jumps to 700 psi again.
- 7:28 Finally holding more or less steady at ~70 psi
- 7:37 Still bouncing around & plugging.

~12:00pm - V3 toggle switch came off.
This had to be repaired before
Condition 7 could be ~~done~~ started.

~2:00 Repaired V3 - running 7 / Decided to do highest gpm
@ 30 psi for 7

~3:30 Rheology taken - ~~Fluxing~~  Because optimum was
4.2 gpm @ 50 psi -
but had already been done 2 times.

* When rinsing Cup added ~30 extra
ml DI to CUP

7:00 pm Sampled - 001 & 002 @ Sec. 5.3.4 in test plan.

6:45pm back pulsed 2 times / Started De watering
target 4.2 gpm / 50 psi - Actual 3.5 gpm / 50 psi.

7:16pm Finished De watering

Project No.  Date of Work 1/10/99
Entered By  Date 1/10/99
Disclosed To and Understood By _____
Signed 1. _____ Date _____

AZ-102 HOT CUF

8:30pm Finished adding all additional material to the CUF.

Recovery solids visually ~1050 settled solids
~9050 Supernate

Ralph used Supernate to rinse other samples into CUF.

When addition was complete noted no movement in the CUF and no flow rate -

The system was plugged.

~9:30 ~45 minutes of manual start and stop (bump the pump) action was required before the system would run continuously. An additional 45 min of running and bumping were needed before de-watering could be started.

* See notes from Composite & Sampling from this date.

10:50 De-watering Complete - Slurry is too thick and stagnant in zones - See video -

Added ~50 ml permeate back - still stagnant
then added ~100-110 ml permeate back - mostly flowing but not uniform
then added ~50 more back - smooth flow (uniform)

~~Started~~ Took Slurry samples
~11:40-11:45

~~Start~~

11:51pm Started 2nd test matrix. Cannot reach target cross flow velocity.

1/11/00 1:15 am

Started condition 2

2:35 am

The slurry appears to not be mixing - added 102.3g DI water to improve mixing. Volume may be lower than before - hard to tell. (cross flow velocity)

Project No. _____ Date of Work 1/10/00
Entered By [Signature] Date 1/10/00
Disclosed To and Understood By _____
Signed 1. _____ Date _____

AZ-102

2:50

The flow appears to be a constant. Higher pressures (up to 30-40) down to 0 psi all produce ~2.5 gpm. Increased air flow doesn't appear to help.

At 60 psi air feed & 30 psi ΔP , we achieved 2.4 gpm w/ Air booster at ~80 (70-90 psi pulsing), with 30 psi ΔP we achieved 2.4 gpm.

Tare weights of sample ~~bottles~~ ^{flasks} Vials.

7 - 16.926g

8 - 16.893g

9 - 17.004g

3:30

Took filter off back end of CUF air motor to increase flow. Maybe 0.3 gpm increase.

4:14

Started condition 4

5:22

Skipped condition 5

5:24

Started condition 6

5:47

Something happened. Pressure jumped to ~100 psi momentarily we then reduced the pressure and the flow rate jumped to 1 gpm more. The filtrate flux decreased by more than half. The pressure drop across the filter went from 2-4 to ~7-8 psi. The flow rate is 3.3 gpm as compared to 2.5-2.7 gpm before.

6:43

Running ~~mat~~ ^{flask} test at 30 psi & 3.7 gpm since suddenly we can get there.

AZ-102 WW Permeate #1
" " " #2
" " " #3

Tare WT.

100.00g
100.29g
100.58g

Project No. _____ Date of Work _____

Entered By _____ Date _____

Disclosed To and Understood By _____

Signed 1. _____ Date _____

K6R

AZ - 102

10:40

Washing Paul's apparatus.

11:45a

Finished washing viscometer. Added 1st water wash solution.
Beginning permeate removal of wash. Lost ~10 ml total.

11:55a

Added oil to oiler for pump.

12:00p

going to 50 psi & max flow.
Backpulsing. Twice.

12:35p

beginning ww2. Backpulsing twice.
Add water. Initiate permeate removal. Backpulse. Take flow rate.

1:35p

done w/ ww2.
Added ww3. Backpulsing.

3:15p

done water

3:30p centrifuge tube. 6.4899g

Empty water wash bottles.

#1 102.8602 g

#2 102.9535 g

#3 102.9225 g

Centrifuged sample 8.6 ml

Centrifuged solids 1.6 ml

~ Volume of slurry calculated to be 865 ml @ this point (K6R Rep)
(calculator)

See next page for NaOH calculations

* Large vessel Calibration (K6 Brooks)

< 750 - not linear

7/8" = 750 ml

1 3/4" = 1400 ml

2 1/2" = 2000 ml

Project No. _____ Date of Work _____

Entered By _____ Date _____

Disclosed To and Understood By _____

Signed 1. _____ Date _____

AZ-102

Caustic Leach Calculations

$$3 \times \frac{V_{\text{solid}}}{V_{\text{total}}} \times V_{\text{slurry}} = X \text{ volume NaOH}$$

$$3 \times \frac{1.6}{8.6} \times 865 = 483 \text{ ml}$$

$$V_{\text{slurry}} - \frac{V_{\text{solid}}}{V_{\text{total}}} \times V_{\text{slurry}} = Y \text{ volume liquid target @ } 3M \text{ NaOH}$$

$$865 - 160.9 = 704 \text{ ml}$$

$$\frac{3 \text{ M NaOH}}{1 \text{ L}} \left(\frac{X+Y}{1187} \right) \text{ L} = \text{moles NaOH needed}$$

$$= 3.561 \text{ moles NaOH}$$

∴ 483 ml of 7.37 M NaOH needed to add
as Caustic Leach
NaOH make-up (KP Brooks)

142.50g NaOH powder
483 ml H₂O

Tare bottle & lid = 66.93g

Bottle, lid & Soln = 676.49g

609.56 g of 7.37 M NaOH were prepared
for Caustic Leach in CUF.

6:25 pm
1825-

Slurry Sample CUF-AZ-102-007 taken

The Sample Port. was removed from the CUF to
attach the hose for transfer of the Slurry to the
Leaching Vessel. It was rinsed with ~10 ml DI into
The leaching vessel.

✱

Problem arose with vessel lid - it is too small
and baffles are too short. Another was fabricated
before Slurry transfer to vessel.

Project No. _____

Date of Work _____

Entered By _____

Date _____

Disclosed To and Understood By _____

Signed 1. _____

Date _____

AZ - 102

2152 All is set and starting of leach/heat cycle
1/11/00 T = 0 Temp = 24 °C begins.
2153 pm

Time = 2157 Temp = 34 °C

1/11/00	Time	Temp C	Time	Temp C
	2159	39	0305	88
	2201	47	0330	85
	2202	50	0405	87
	2206	60	0431	86
	2209	70	0500	84
	2214	83	0530	82
	2215	85	0600	83
	2220	88	0628	86
	2223	88		
	2224	87		
	2230	85		
	2234	82		
219	2254	85		
	2302	82		
	2317	86		
	2321	84		
1/12/00	2346	85		
0005	88			
0031	85			
0100	88			
0132	84			
0201	85			
0232	81			

Controller Calibration Expires 8/2001
S/N 2013

Pat TC TC-325-426 - 8/00 Expires

Rinsed out "AZ-102 Caustic Addl" it will replace

"AZ-102 CL permeate".

387.321g DI water container initial

Project No. _____ Date of Work _____

Entered By _____ Date _____

Disclosed To and Understood By _____

Signed 1. _____ Date _____

Date _____

AZ-102

361.782 g DI water after rinsing off the impeller

25.539 g DI water added

6:55 Weighed & measured level on container

7:07 Added slurry to CNF, Turned on heat tape

7:27 Rinsed ^{leach container} ~~CNF~~ with DI water7:30 $T_{\text{CNF}} = 58^{\circ}\text{C}$ 7:35 $T_{\text{CNF}} = 67^{\circ}\text{C}$ 7:42 $T_{\text{CNF}} = 75^{\circ}\text{C}$ 7:46 $T_{\text{CNF}} = 79^{\circ}\text{C}$ 7:50 $T_{\text{CNF}} = 83^{\circ}\text{C}$ 8:00 $T_{\text{CNF}} = 88^{\circ}\text{C}$ KGR
1/12/008:22 Dewatered caustic leach.
Acquired -008 & -009 after dewatering.

8:09:17 Tare wt's

CNF-AZ-102-010	16.988g
-011	17.008g
-012	17.023g

10:27a 596.58 g - wt. of caustic leach permeate removed. Total. Bottle & solution,

wt. of 1st c.w. solution w/ bottle.
1659.86g
bottle 267.63g

Project No. _____ Date of Work _____

Entered By Kenneth R. [Signature] Date 1/13/00

Disclosed To and Understood By _____

Signed 1. _____ Date _____

2. _____ Date _____

116 R
1/12/00

AZ 102

Drained slurry. 10:55a. level in washing vessel. $13\frac{1}{16}$ -inch 101

Tare wts.

CUF-AZ-102-013	16.861 g
-014	16.969 g
-017	16.913 g
-016	16.803 g

11:40p Drained caustic wash solution into vessel. Draining pump housing & blowing out upper loop. Draining pump housing again.

Rinsing out drain container. Determining how much rinse we add.

tare wt. 145.000g w/wash 154.500g
added 9.500g wash

Level $2\frac{7}{8}$ -inches in leaching container.

3520.6g slurry in leaching vessel.

12:30p Heating caustic wash vessel. Now @ 40°C.

<u>Time</u>	<u>Temp (°C)</u>
12:30p	43
12:40p	62
12:50p	82
12:54p	85
13:25	86
13:56	87
14:26	88
19:42	87
21:00	84

21

9:00 pm Heat time complete -

* Manipulator hand/band snapped - Working with one manipulator
Cannot pull samples from "middle" of dewatering.

Project No. _____ Date of Work 1/12/00

Entered By [Signature]

Date 1/12/00

Disclosed To and Understood By _____

Signed 1. _____

Date _____

2. _____

Date _____

AZ-102

Slurry successfully transferred into CUF

Started @ ~ 65 °C

2153 Temp up to 74.3

2225 - Temp @ 88 - Working on manipulator repair

Climbed to 92 - turned on chiller for 1 min
dropped back to 85

2328 T = 79.7 Still working on manipulator repair.

0054 Started dewatering

1:03 Stopped dewatering & pulled samples.

Transferred the slurry to the Pot

Made up some more 0.01 M NaOH - needed 300 ml

Added 0.12 g of NaOH pellets to container to
make 1400.7 g total weight of liquid. (already had
1100 ml of 0.01 M NaOH from the rest of the lot)

0.010 M NaOH

10X dilution of standardized 0.1M NaOH

7.0 Liters prepared

Expiration date 1/1/2001

RE: Chemres_37 —rgs

LAB 13745 p.102

This label was for all of the other 0.01 M NaOH used during the
Washes. ~~etc~~

2:35 Add water to CUF & mixed around

6:52 Transferred liquid into pot

Project No. _____ Date of Work _____

Entered By _____ Date _____

Disclosed To and Understood By _____

Signed 1. _____ Date _____

2. _____ Date _____

Discovered that the heat tape was running continuously during the last run - last ones to start it hook it directly to power.

Pot has been uncovered for the last 30 minutes losing $\sim 0.01\text{g/sec}$ to evaporation

3:16 Put on lid

4:02 Started stirring

4:13 Reached 85°C

4:30	84°C
5:00	83°C
5:35	83°C
6:02	83°C
6:30	82°C
7:01	85°C
7:30	84°C
8:00	83°C
08:30	86°C
09:00	87°C
09:30 ^{by 1-13-00}	88°C
10:00	87°C
10:00	87°C
10:30	84°C
11:00	82°C
11:30	82°C
12:00	83°C
12:12	shut down

12:13p Mixing & heating stopped. Disassembling CUF wash vessel.

* Significant solids stuck to mixing baffles.
Attempting to scrape off.

Project No. _____ Date of Work _____

Entered By Lennell R. Goff Date 1/13/00

Disclosed To and Understood By _____

Signed 1. _____ Date _____

2. _____ Date _____

K6R
1/13/00

** significant solids also stuck to mixer.
Will attempt to rinse off.

Squirt bottle before rinse : 432.51 g
after rinse : 332.442 g
100.068

Rinsing appeared successful aside from some
crusted-on material on stems of impeller.

Weighing pot. 3237.10g

Rinsing pot. Twice. Large chunks did not come
out after initial pot dump.

Additional water added: bottle 182.35g
 $332.442 - 182.35g = 150.092g$

01:30p everything in CUF, heating.

Time	Temp
1:30p	61°C
2:06p	76°C
3:00p	83°C

(not increasing)

wt. of empty
leach vessel.

1136.78g

12.37g slurry * water lost

Need weights of: Empty caustic wash permeate #2 bottle
Tare wt. of CUF-AZ-102-015

Need samples of Water Wash Permeates #1, #2, & #3. 2 each.

Tare wt of CW permeate #2 bottle: 261.982g

Tare wts of:	CUF-AZ-102-015	16.714
	-018	16.951
	-019	16.846
	-020	16.924

Project No. _____ Date of Work _____

Entered By Kenneth A. [Signature] Date 1/13/00

Disclosed To and Understood By _____

Signed 1. _____ Date _____

2. _____ Date _____

166P

1/13/00

Tare wts.	CUF-AZ-102-021	16.8810
	022	16.5307
	023	16.9559
	024	17.0717
	025	17.0804
	026	17.1539 41

3:05p Setting CUF to 30 psi & max cross-flow.

Flowrate initial

3:12p 1/13/00 4.59 sec. 20 ml 1.9 gpm 28 - 30 psi

3:30p 85°C reached, Beginning de-watering.
Half-complete

3:45p Acquired samples CUF-AZ-102-021 & -022.

3:50p Resume dewatering. Done. Filling ~ 100 ml extra due to additional water added for rinsing wash vessel.

Sample weights.

CUF-AZ-102-021	35.402g
-022	37.734g

** Filtrate appears quite bubbly, like possibly getting a lot of foaming.
Rheology vid for Paul's final sample. 5.3.72.

tare: 133.3506g
filling with ~ 80 ml of slurry.

** Heat tape controller has been going screwy. Realized slurry temp. had dropped to < 70°C. Heating back up.
Saw T.C. line was getting slightly charred by the heat tape.

4:30p Pump shut down - no flow.
Looked in slurry tank, no mixing, way too thick.
Got ~~about 40 ml~~ ~40 ml of sample for Paul's rheology.

Project No. _____ Date of Work _____

Entered By Therese Kays Date 1/13/00

Disclosed To and Understood By _____

Signed 1. _____ Date _____

2. _____ Date _____

AZ-102KAR
1/13/00

Tried restarting pump. No flow. Shut back down.

Tare wt. of Caustic W. P. #1 bottle. 102.8196g
303.6570gAdding 200.8374 g Δw.AG
1/13/00

5:20 pm Slurry is pumping again but visual show a thick coating of Solids all along the walls of the CUF Tank.

Attempting to scrape the walls down before proceeding.

Temp ~~of~~ of Slurry was ~47°C. Turned on chiller.

5:35 T = 20.6°C

Sent in 3 bottles - 1. Large Plastic Coated (~1 quart) Tare
"AZ-102 Final Sample" → 520.37
N300ml plastic Coated → 354.756g
"AZ-102 Washed Solids 1st Rinse"
1 L plastic polyethylene
AZ-102 CUF 1st Rinse → 105.575g

5:55 Scraping sides did not return any solids to slurry but recover some to be recovered in Rinse.

Taking Rheology Sample
Final wt. 192.540 g

lost ~5g from spout -

4 Slurry samples taken

Project No.

Date of Work

Entered By

Date

Disclosed To and Understood By

Signed 1.

Date

2.

Date

1/13/00

1/13/00

1

1

0:50 First rinse
Weight of DI Beak @ start 542.93 g

Washing down tank side

DI bottle after washing 63.179 g

DI 479.7502 g added

7:10 - CUF on allowing to mix.

7140 - Dried into ~500 ml Glass - A2-102 washed solids 1st time
318.284 g removed

Part in 1L DI H_2O from chem add task.

AZ 102-CUF Ring #1 $\frac{1040.26 \text{ g}}{105.575 \text{ g}} = \text{full}$

934.685 g Sherry in
bottle
removed from
cwt.

Sent in ^{5 Ad} ~~three~~ more 1L Poly bottles
Rare

AZ 102 CVFR. nsc #16 $\frac{1 \text{ sec}}{102.89}$ $\frac{1 \text{ min}}{405.16 \text{ g}}$

AZ 102 CUF Ring #2 102.97 1065.97

AZ-702 CUF Ring #2 b 102.94 g 714.54 g

9:50 AZ-102 COF Ring #3 $\frac{100.016}{g}$ $\frac{1069.41}{g}$

✓ 16 AZ-102 CUF Riser #4 $\frac{100.551}{100.551} \times 1037.09$

9:00pm - Ralph noted U4 maybe failing

11:30 - Hooking up cartridge -

Project No. 11-11-1 Date of Work 1/13/80

Entered By [Signature] Date 1/13/00

Disclosed To and Understood By

Signed 1. _____ Date _____

1/14/00

ATB

1:03 am started the 0.05 μ m filter cleaning the CUF
(external filter)

2:21 Backpulsed twice — very slow!
Backpulsed again

2:30 Drained system — still kind of brown

2:40 Refilled w/ water (1 l) attached filter & ran

Checked water exiting filter — very clear
Checked water out of V9 — still very yellow
Filtrate flow rate is very slow — clean water fluxes not recovered.
Pulled off supernate from A2-102 final material. Picked
up some solids — will have to filter.

4:45 Backpulsed ~ 600 ml of HNO_3 thru the top

5:06 Turned on CUF w/ 1 l of 1 M HNO_3

6:00 Drain 1 M HNO_3 — muddy brown colored

Put ~ 600 ml of 1 M HNO_3 / 0.2 M Citric
into CUF

6:30 Removed acid & replaced w/ 1.5 l of DI
water

The acid is lighter this time, but still fairly brown

7:00 Backpulsed several times and drained liquid. Still
fairly brown.

7:12 Backpulsed 3 thru the top & drained the liquid

7:20 Added second batch of water — running thru 0.05 μ m filter

Project No. _____ Date of Work _____

Entered By _____ Date _____

Disclosed To and Understood By _____

Signed 1. _____ Date _____

2. _____ Date _____

1/14/00
K6R

109

12:00p rinsing rheology equipment.
Everything to go into 'AZ-102 Final Sample' jar.

DI water bottle weight.

Before rinsing: 509.62g

After rinsing: 399.22g

Rinsed with 110.40g DI water.

Good recovery of sample !!

Final Sample jar weight:

'AZ-102 Final Sample' = 1254.17g

Checking pH of second rinse. Done. have no standards, so leaving in cell.

Checking water filtrate flux measurement.

30 psig
** Max pressure attainable, just < 40 psig.
Therefore, just running @ 30 psig * 40 psig.

2:20p Backpulsed once upon completion of water tests.
Opened V2 & V10 to flow through external filter cartridge. Ran pressure up to ~ 25 psig for ~ 15 minutes.

2:35p Backpulsed twice. Drained.

* Looks clear.

1/11/00 * Note Clean water fluxes not achieved At 1/14/00



Note Visually Solids have settled well in final sample & washed solids rinse #1 - but in CUF Rinse 1 - 4 not yet settled enough to recover. ∴ leaving All till Monday. Additionally - based on the settling in the high solids jar, a lot of air was entrained in the slurry when removed from CUF.

Project No. _____ Date of Work _____

Entered By K6 Rappé Date 1/14/00

Disclosed To and Understood By _____

Signed 1. _____ Date _____

2. _____ Date _____

Appendix B: Testing Mass Balance

Appendix B: Testing Mass Balance